NASA CR-_____

SEMIANNUAL REPORT

DEVELOPMENT OF FUEL CELL ELECTRODES

by

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prepared for

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CONTRACT NAS 3-9430

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SECTION I

1.0 SUMMARY

The Report describes the progress made under Task 1 of NAS3-9430 during the period from January 1, 1967 to June 30, 1967. During this period 36 hydrogen-oxygen fuel cells were tested with circulating KOH electrolyte. The majority of these tests were at 200 ASF and 90°C, although limited tests were conducted at 100 ASF, 300 ASF, and on a simulated NASA duty cycle involving periodic power cycling. In nearly all of these tests various modifications of the LAB-40 electrode were used as cathodes, while both LAB-40 and T-2 electrodes were tested as anodes. Two sizes of cells were evaluated: a) a "small" stack containing $1/8$ ft^2 of active electrode area, and b) a "flight-size" stack containing 1.3 ft^2 of active electrode area. Testing at 200 ASF was conducted both at 15 psia and 30 psia. Typically increasing the pressure from 15 to 30 psia resulted in about a 40 mv increase in cell potential. Of the 15 small cells tested at 200 ASF and 15 psia, 10 met one of the two program milestones—namely, generation of an initial potential of 0.90 volt at 200 ASF. From the same group 8 stacks met the second program milestone of a potential decay rate of less than 20 mv in 500 hours. One-third of the cells met both milestones. Two stacks from this group operated for more than 2000 hours.

Six small cells were tested at 200 ASF and 30 psia. Of this group two suffered mechanical failures on startup. The remaining four all met the initial voltage milestone and two also met the voltage degradation milestone. The two tests which did not meet the voltage degradation milestone were terminated because of test stand operational problems rather than potential decline.

Four flight size cells were tested; two at 15 psia and two at 30 psia. All four met the voltage decay milestone, and the two 30 psia tests also met the initial potential milestone. Excellent performance of those cells operating under the similated duty cycle was observed. On the basis of test results accumulated during this period the LAB-40 and T-2 electrodes are comparable as anodes.

Initial attempts to modify the Union Carbide T-3 cathode for high noble metal loading have yielded encouraging results in small cells but several processing variables must yet be explored before an optimized fabrication procedure is established.

2.0 INTRODUCTION

This is the second Semiannual Progress Report to be written under Contract NAS3-9430, and covers work completed under Task 1 during the period January 1, 1967 to June 30, 1967. As specified in the contract Work Statement the purpose of Task 1 is to conduct a program to improve the performance capability of the Union Carbide thin fixed-zone electrode. Task 2 of the contract involves preliminary design of a 5-kw hydrogen-oxygen fuel cell power supply. This task was completed during the present report period, and the work is described in detail in a separate Topical Report.

Work during the past six months has been channeled into three major areas. A major fraction of the effort has been performance testing of those promising electrodes identified during the first six months of work. Basically the testing has involved "hybrid" cells using Union Carbide Type-2 anodes and American Cyanamid LAB-40 cathodes or cells using the LAB-40 electrode as both anode and cathode. Several modifications of the LAB-40 backing have been tested in an effort to obtain electrodes which do not delaminate during service or leak KOH into the gas spaces. An essential and integral part of the testing work has been to provide test stands capable of reliable operation at both 15 and 30 psia and 100°C. All tests have been with a circulating KOH electrolyte—a key design concept in Union Carbide Fuel Cell systems.

The second major work area has involved studies on a modified Union Carbide Type-3 electrode. The modification under investigation is replacement of most or all of the carbon used in the commercial version of the T-3 electrode with platinum to achieve performance levels of importance to NASA. Emphasis in this work has centered on the screening of various electrode processing variables using subsize electrodes.

The third work area to which limited amount of experimental effort has been devoted is the screening of possible cell construction materials to assess their compatibility with 12 N KOH at 100°C. Testing has emphasized various thermoplastics and elastomers.

SECTION III

3.0 FACTUAL DATA

3.1 Electrode Studies.

This section describes development work on platinum-loaded Type-3 electrodes. The Union Carbide Type-3 (T-3) cathode consists, typically, of a dual-porosity metal substrate with a coarse, large-pored, metal layer bonded to a very fine-pored layer. The electrochemically active material is contained in the coarse layer, and may be backed by an additional electrolyte-repellent layer as well.

In operation the fine-pored metal layer is in contact with the electrolyte, and is completely wetted through. The large-pored layer containing the active material is maintained in the proper state of semiwetness in part by means of the binder and wetproofing agent employed (e.g., Teflon), and also by the application of sufficient gas pressure to force the bulk of the electrolyte out of the coarse-pored region. However, the gas pressure is insufficient to force the KOH through the fine-pored layer. The electrolyte-repellent backing provides additional protection against electrolyte leakage, and may be particularly useful during standby and startup when it is possible that no back pressure will be present. A further advantage of this repellent layer is the safety it provides against nonuniformity in the electrode structure which might be present in the form of isolated defects constituting points of leakage.

During this reporting period effort has been directed towards development of a modified T-3 cathode which is loaded with platinum at about the 40 mg/cm² level. Evidence to date indicates that current-voltage characteristics (IR-free) equalling those obtainable with LAB-40 cathodes can be achieved. Thus far, processing variables have been examined only in sufficient depth to delineate the direction for future experimentation.

3.1.1 Experimental Approach.

Because of the high cost of platinum experimental electrodes were made in 2" x 2" size. In turn, these were cut into 1-inch squares and tested in small cells as described previously[1]. Although Union Carbide had

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(1) NASA Contract Report (CR-72178); Semiannual, Period: June 30-Dec. 30, 1966; Contract NAS3-9430, p. 7

previously developed a substantial amount of information on a commercial version of the T-3 electrode with very low Pt loadings, no work had been done on a high-platinum version for NASA applications. Consequently, to provide a suitable standard for comparison it was decided to screen some of the fabrication variables on Type-5 electrodes on which some experience with higher Pt loadings had already been obtained during the previous reporting period[2]. Although useful information was obtained in this manner, particularly with respect to such variables as cure temperature, binder type, and binder amount, the T-3 structure is sufficiently different that the results were not always directly transferable from one type of electrode to the other.

3.1.2 Experimental Results.

Platinum black obtained from Engelhard Industries (90-100 A° crystallite size) is being used exclusively at the present time. However, some of the earlier work was done with platinum black precipitated from platinum chloride in our laboratory (designated as UCC Pt), and certain fabrication variables were tested with this material. While this established useful trends, the differences in physical properties between these two types of platinum will require additional work to conclusively establish optimum electrode fabrication procedures. A summary of results which are considered useful in directing future work follows, and is presented in tabular form in Table I.

3.1.2.1. Results with T-5 Cathodes:

a. Binder Type: Both TFE and FEP Teflon have been found to be usable as binders. Tests 1 through 4 (Table I) give examples obtained after two days on test at 200 ASF, 80°C, in 12 N KOH. Since the screening of variables involves a large number of tests, life testing of small samples has not been undertaken. Rather, the test-stand capacity has been devoted to analysis of processing variables. Thus, electrodes are run for only a week or two. For initial screening purposes, potentials taken after two days of operation are used for comparison. This provides an initial break-in period but disregards subsequent voltage decay, and thus probably represents near peak performance of the electrodes. Tests to date do not conclusively indicate superiority of either type of Teflon.

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(2) NASA Contract Report (CR-72178); Semiannual, Period: June 30-Dec. 30, 1966; Contract NAS3-9430, pp. 22-24.

TABLE I

EXPERIMENTAL RESULTS WITH ALL-PLATINUM T-5 CATHODES[1]

Test No.	Pt Type	Binder	Processing Pressure (psi)	Cure Temperature (°C)	IR-Free Potential (v) vs. Hg/HgO Ref.[2] After 2 days
1	Union Carbide	10% TFE	100	275	-0.005
2	Union Carbide	10% FEP	200	200	-0.002
3	Engelhard	10% FEP	100	200	-0.020
4	Engelhard	25% FEP	100	200	-0.001
5	Union Carbide	10% TFE	100	200	-0.012
6	Union Carbide	15% TFE	100	200	-0.004
7	Union Carbide	10% TFE	100	275	-0.043
8	Union Carbide	15% TFE	100	275	-0.012

(1) All tests run at 200 ASF in 12 N KOH at 80°C.
(2) LAB-40 cathodes are usually in the +0.010 to +0.020 volt versus Hg/HgO.

b. <u>Binder Amount</u>: The Teflon:Pt ratio has not been studied exhaustively. Tests 3 and 4 (Table I) give examples of two FEP binder levels; tests 5 and 6 of two TFE levels. Although these results suggest somewhat better performance at the higher Teflon percentages, data are not adequate to draw firm conclusions.

c. <u>Curing Temperature</u>: Electrodes have been baked at temperatures ranging from 200 - 300°C, and although the results again are inconclusive they appear to favor the lower temperatures. Tests 7 and 8 represent pieces of the same electrodes as tests 5 and 6, except that they were cured at 275°C.

3.1.2.2 <u>Results with T-3 Cathodes</u>.

After preliminary testing of all-platinum T-5 electrodes, more extensive trials with T-3 cathodes have shown that they present a different set of problems. These problems are summarized below:

a. Pressures used in fabricating T-5 electrodes are insufficient when making T-3 structures. In the former case, pressures of 100 - 200 psi were used. In the latter pressures in the order of ten times these values are necessary. An optimum T-3 structure must have the coarse-metal layer completely filled with the platinum mix. A metallographic cross section of one of the best T-3 cathodes has shown that even at 2000 psi the mix has only penetrated about halfway through the coarse layer. As a result, electrodes have been quite variable, and have shown undesirable results as a function of the oxygen to electrolyte differential pressure. On the other hand, increasing the fabrication pressure to above 2000 psi has resulted in poorer electrochemical performance in some tests, apparently by decreasing gas permeability to a small value.

b. Even though very encouraging results have been obtained when a small amount of acetylene black is used as an extender in the T-3 electrode, omission of the carbon leads to inferior performance.

c. Although results with T-5 electrodes indicate the possible benefits of lower curing temperature, most T-3's have been cured at 275°C. This is because the electrolyte-repellent layer being used at present is a high TFE-bonded inactive carbon layer. Although 275°C is considerably below the optimum TFE cure temperature, it is felt that a higher temperature is probably incompatible with good cathode performance (i.e., probable detrimental effect on platinum catalyst). Further work needs to be done to establish this point.

The fact that it is possible to approach the electrochemical performance of LAB-40 electrodes is shown in Fig. 1 where results of three T-3 cathodes are plotted. Test A was started at 2 psig back pressure and behaved erratically. After four days, the oxygen back pressure was reduced, and the cathode potential immediately increased to a value in the LAB-40 range where it held until the test was discontinued to make room for other tests. Test B was started at 0 psig back pressure, and was increased to 2 psig after four days on test; this resulted in a small improvement.

An additional problem with T-3 cathodes, which can only be evaluated in larger cells, is the question of electrode resistance as evidenced by IR drop in the cell. Earlier tests with cells containing T-2 cathodes indicated a probable oxidation of the porous nickel and/or active carbon which resulted in a progressive increase in IR drop. Present T-3 cathodes are made with gold-plated, porous Ni; this has proven not to be totally satisfactory. The plating procedure has not resulted in much more than surface coating of the porous nickel; a 1/8-sq. ft. cell (A-095) also showed progressively increasing IR drops.

Cell A-095 was the only cell with T-3 electrodes tested to completion during the present reporting period. It performed poorly, and fell well below acceptable NASA standards. This, in part but not entirely, was the result of inadvertently applying too high a pressure during fabrication. Because of the steep current-voltage curve, life testing was conducted at 100 ASF. The cell was run for ten days at this current density with the terminal voltage ranging from 0.84 to 0.92 volt. Sensitivity to differential oxygen pressure was noted. At a pressure differential of 25" w.c., the voltage declined from 0.89 to 0.84 volt in three days. Upon increasing the ΔP to 65" w.c., it improved to 0.92 volt and then declined to a final value of 0.86 volt.

Figure 2 represents both IR-free and IR-included cell voltages taken at two different times. These curves illustrate the IR-drop increase previously mentioned, and show that a significant part of the voltage decline was due to internal resistance.

3.2 Stack Testing.

3.2.1 Facilities.

Under the terms of Contract NAS3-9430 Union Carbide Corp. agreed to provide 18 stack test stands, of which 12 were to accommodate small

Test Conditions:
200 ASF
12 N KOH; 80°C
Engelhard Pt(100 A
Units-Crystallite
Size) + Acetylene
Black
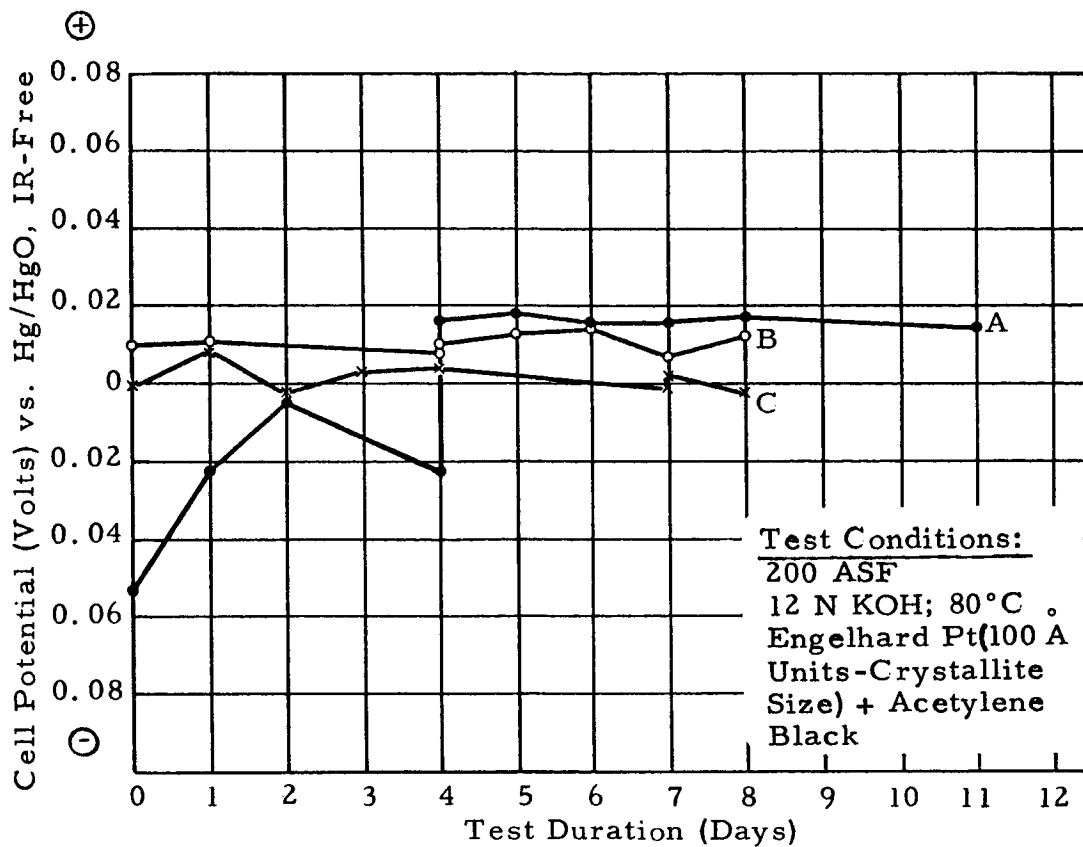
A = 4 to 0 psig; B = 0 to 2 psig; C = 0 to 2 psig.   D-3467

Fig. 1 - Performance of T-3 Cathodes.
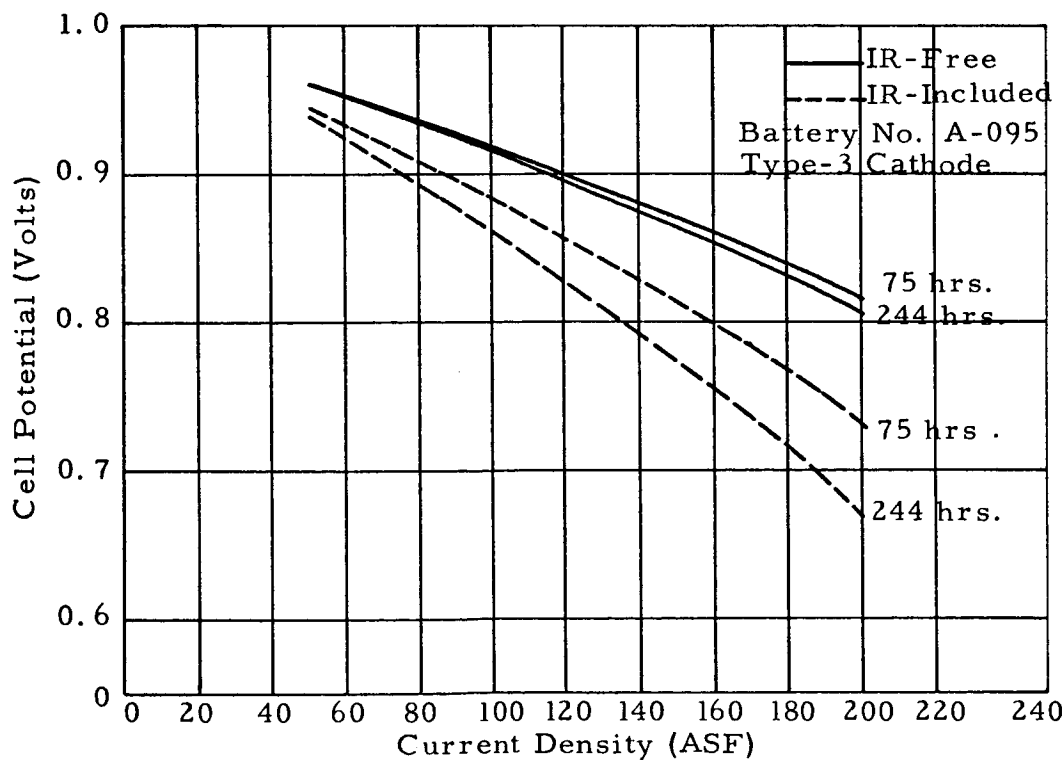
D-3466

Fig. 2 - Polarization Curves for Cell A-095.

cells (about 1/8 ft² electrode surface) and six were to take so-called flight-size cells which were to have at least 1 ft² of active electrode surface. This commitment required fairly extensive modification of the existing test facilities and considerable new construction, because the smaller size is not widely used in normal Union Carbide programs, and the larger size, though generally used, is not usually operated at the high current densities required here. Furthermore, normal Union Carbide operations are carried out at or below 70°C, which permits the use of polypropylene electrolyte tanks and plumbing while the 100°C requirements of this program necessitated the use of nickel as the construction material.

Initially six small test stands were activated and used in the early stages of the program. An additional six small test stands, more highly instrumented and capable of more careful control and monitoring than the first group, were completed largely during the winter months. Both designs have already been depicted and discussed in the first Semiannual Progress Report (NASA CR-72178, pp. 8-14).

Subsequently, six large test stands for the flight-size cells were built. Of these, four were for operation at 15 psia (normal atmospheric pressure) and two for operation at 30 psia or higher. The 15 psia stands are substantially similar to the small stands except for such obvious modifications as larger electric loads and leads, larger fluid ducts and meters, and larger water collection devices. The large 15 psia stands are shown in Fig. 3. The 30 psia stands are depicted in Figs. 4 and 5. The large test stands could also be used for small cells as the situation required, in which cases the electrical systems were modified.

3.2.2 Test Procedures.

Most of the tests described in this report were life tests at 15 psia conducted on small cells. For these, the following procedures were used: The electrolyte was 14 N KOH, heated in the electrolyte tank to give a stack outlet temperature of 90±2°C. The nominal electrolyte circulation rate through the cell was 124±10 ml/min. Sometimes this value was exceeded for reasons of safety in cells, wherein the KOH flow rate required a ΔP of only about 1" since the low ΔP was very difficult to maintain. In practice, the KOH inlet pressure was generally 16.5" of KOH; the pressure drop through the cell was 3-4". A determination of the electrolyte flow as a function of pressure drop
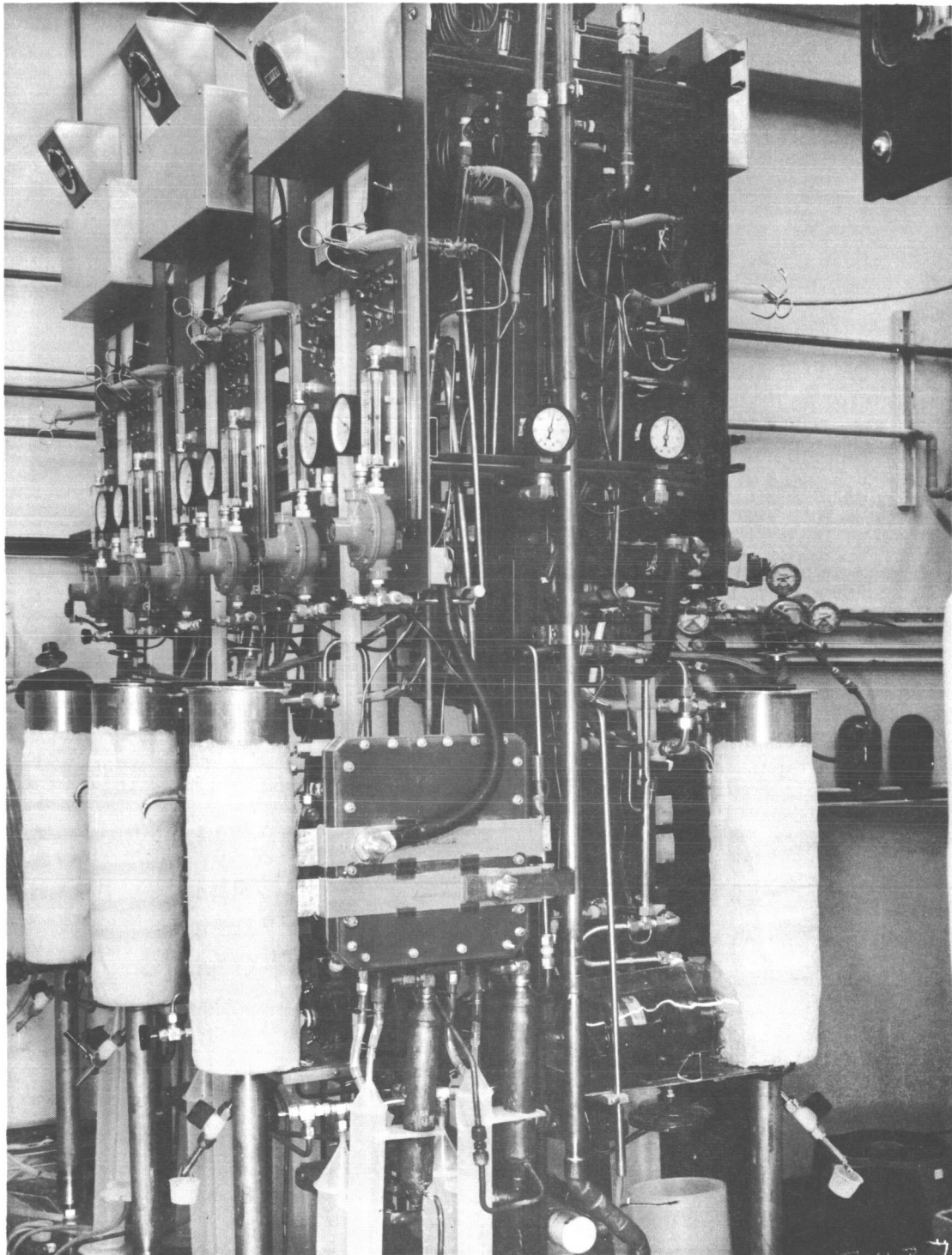
D-3437

Fig. 3 - Test Stands for Flight-Size Stacks Operated at 15 psia.
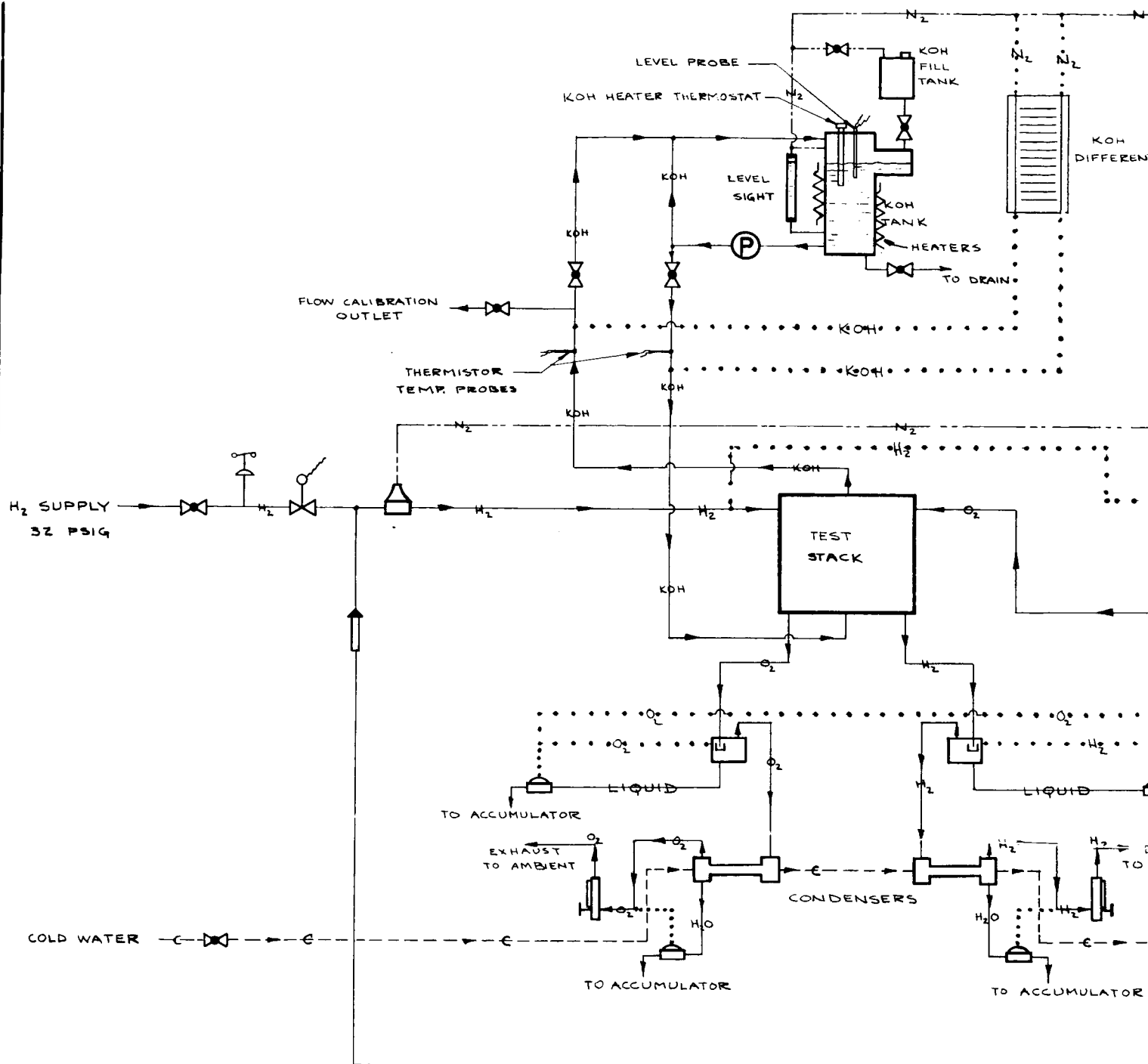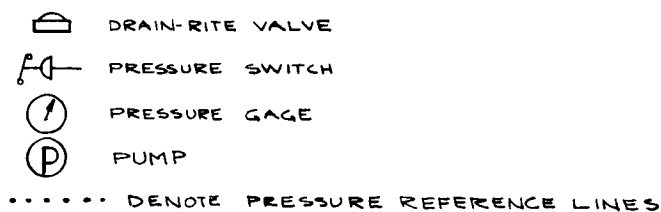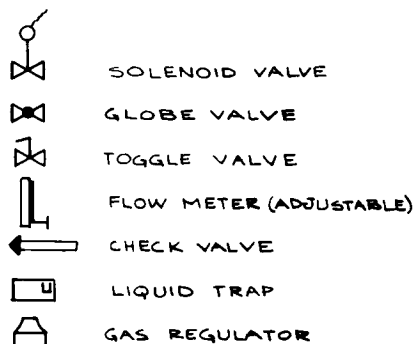
Fig. 4 - Schematic Drawing of

LEGEND

Symbol	Description	Symbol	Description
	SOLENOID VALVE		DRAIN-RITE VALVE
	GLOBE VALVE		PRESSURE SWITCH
	TOGGLE VALVE		PRESSURE GAGE
	FLOW METER (ADJUSTABLE)		PUMP
	CHECK VALVE	......	DENOTE PRESSURE REFERENCE LINES
	LIQUID TRAP		
	GAS REGULATOR		

0.01
0.02 CFH
N₂ PURGE

0-30 PSIG

IAL MANOMETER

N₂

N₂

N₂

N₂ SUPPLY
15 PSIG

O₂ TO N₂
DIFFENTIAL
30 INCHES W.C.

TO AMBIENT

H₂ TO N₂
DIFFERENTIAL
30 INCHES W.C.

H₂

O₂

N₂ O₂

H₂

H₂

O₂

H₂

STACK DIFFENTIAL
2 INCHES W.C.

STACK DIFFERENTIAL
10 INCHES W.C.

N₂

O₂

O₂

O₂ SUPPLY
32 PSIG

H₂

O₂

TO ACCUMULATOR

XHAUST
AMBIENT

ARGON SUPPLY
25 PSIG

- - - - - TO DRAIN

Test Stand for 30 psia Operation.

D-3446

Fig. 5 - Test Stand for Operation at 30 psia.

was part of the normal start-up procedure and was repeated periodically. Usually, no significant flow degradation was found. The electrolyte flow at a ΔP of 1.5" of hot 14 N KOH is included in most of the performance summaries so that the various stacks may be compared.

Hydrogen and oxygen were not recirculated but instead passed through the stacks once and then were exhausted. The flow rates were adjusted to give water balance, i.e., to maintain the electrolyte concentration at the desired point as far as possible. This proved difficult, and in most instances the electrolyte tended to concentrate, so that water was added from time to time. This explains the recorded fluctuations in electrolyte normality. The gas flows were measured on Brooks air purge meters. For the small stacks these were generally kept at what is equivalent to 0.4 scfh of air for H_2 and 2.0 scfh air for O_2, corresponding approximately to 1.56 scfh H_2 and 1.82 scfh O_2. At 200 ASF a 1/8 ft^2 electrode actually requires 0.40 scfh H_2 and 0.20 scfh O_2, so that for most tests the systems were run at what would correspond to a recirculation rate of 4X use rate on the hydrogen side, and 9X use rate on the oxygen side. These flow rates generally require about 0.3 - 1.0" water equivalent ΔP for O_2 and 0.1 - 0.2" for H_2. Gas inlet pressures were set regularly to be 1" below the blow-through pressure for O_2, and 2" below blow-through for H_2. In addition, the difference between the pressures of the two gases was not permitted to exceed 10". Actual inlet pressures were usually in the 15 - 30" range.

The electrolyte temperatures of 90±2°C at the outlet were measured with a thermistor system from Yellow Springs Instrument Co., Yellow Springs, Ohio. Most test stands were equipped with probes both on the inlet and outlet, and in general the temperature difference between the two was about 2°C, the inlet being higher. The heat lost from the stack was greater than that produced by the electrochemical reaction even after the stacks were insulated with either 3/8" Plexiglas plates over the cell end plates or with Styrofoam. Temperature was maintained by heating the electrolyte tank externally with a heating tape controlled by a Ni-plated Fenwall thermoregulator immersed in the electrolyte tank. Electrolyte pressures were measured in standpipes at the inlets and the outlets.

Current, voltage, temperature, gas flows, and electrolyte concentration were checked daily. Condensed water was removed and measured daily; leakage, if any, was measured as needed. The alkalinity of all liquids collected

was checked and recorded if any was found. In general, polarization curves were run on all cells once a week using a Marko-Kordesch interrupter, unless special situations required more frequent data taking.

The Marko-Kordesch Interrupter [J. Electrochem. Soc. 107, 480-483 (1960)] is an instrument which permits measuring the voltage of a battery or fuel cell stack eliminating the ohmic voltage drop. This is called the IR-free reading, denoted by the letter "F" in all our polarization curves. The polarization curve obtained as usual by taking the terminal voltage was also measured with the same instrument and is shown on the same graphs, unmarked, called the IR-included curve. Thus the two curves may be compared directly.

The stacks operated at 30 psia were first started in the standard fashion at 15 psia, 90°C, and 200 ASF. Particular attention was paid to blow-through pressures and electrolyte flows, especially the latter, which could not be checked after pressurization. Then, with the stack on open circuit, the solenoid controlling the flow of nitrogen—the pressurizing gas—was opened. Nitrogen pressure was applied uniformly to O_2 and H_2 regulators and to the entire electrolyte system, so that all portions of the fuel cell system experienced the rise in pressure simultaneously. The H_2 and O_2 purges were watched carefully and adjusted to maintain the correct purge rate.

As far as data taking was concerned the same procedures were followed and the same data taken as for 15 psia operation except: a) electrolyte flow could not be checked; b) N_2 pressure and purge were checked daily, and c) the KOH normality was checked daily by titration of a small sample, instead of the specific gravity method usually used.

The procedures used for the flight-size cells were basically the same as those outlined above for the small cells with such obvious modifications as having the gas flows, etc. about ten times larger.

Task 2 of the contract calls for the design of a 5-kw system capable of performing under a certain load profile. Four tests were run to establish performance of Union Carbide Fuel Cells under a load profile similar to that specified for the system. The tests are discussed below (in Section 3.2.5.3). They differed from all other tests in that the electrolyte was only 11.5 N KOH. All liquids removed from the stack were collected in just two bottles, one in the anode-exit side, and one in the cathode exit. The normality of the liquid was

determined, and the equivalent electrolyte leakage, if any, calculated therefrom. Gas inlet pressures were checked daily. The pressures were set at 1" below blow-through pressure at a fairly low current (ca. 60 ASF), and were not changed, even though the pressure dropped (for the same gas flow rate) at the higher current densities. These tests were carried out on the older, less highly instrumented, group of test stands.

The actual load cycle used in the tests differs in detail from the load cycle specified for the 5-kw system. It has the same percentage of the operating time under the same loads as the system specification requires, but the exact timing was rearranged for convenience of single-shift operation. The following cycle was followed five days each week; operation on the weekend was at 124 ASF.

TABLE II

SIMULATED NASA DUTY CYCLE

From	To	Current (amps)	Current Density (ASF)	Duration (Hours)
0830 hrs	0930 hrs	7.5	63	1.00
0930	1015	14.8	124	0.75
1015	1030	24.0	200	0.25
1030	1130	14.8	124	1.00
1130	1330	7.5	63	2.00
1330	1430	14.8	124	1.00
1430	1630	24.0	200	2.00
1630	0830	14.8	124	16.00

3.2.3 Stack Design.

3.2.3.1 Small Stacks for Use at 15 psia.

A typical small stack for use at atmospheric pressure is depicted in Figs 6 and 7. The specific materials used which varied in different stacks are itemized in Table III. The active area of each electrode in this design is nominally $1/8$ ft^2, with the electrodes separated from each other by the electrolyte separator specified in Table III. Initially, an expanded-nylon separator was used, but autopsy of several long-lived cells after failure showed that the nylon separator had disintegrated. This permitted the electrodes to touch and short out.
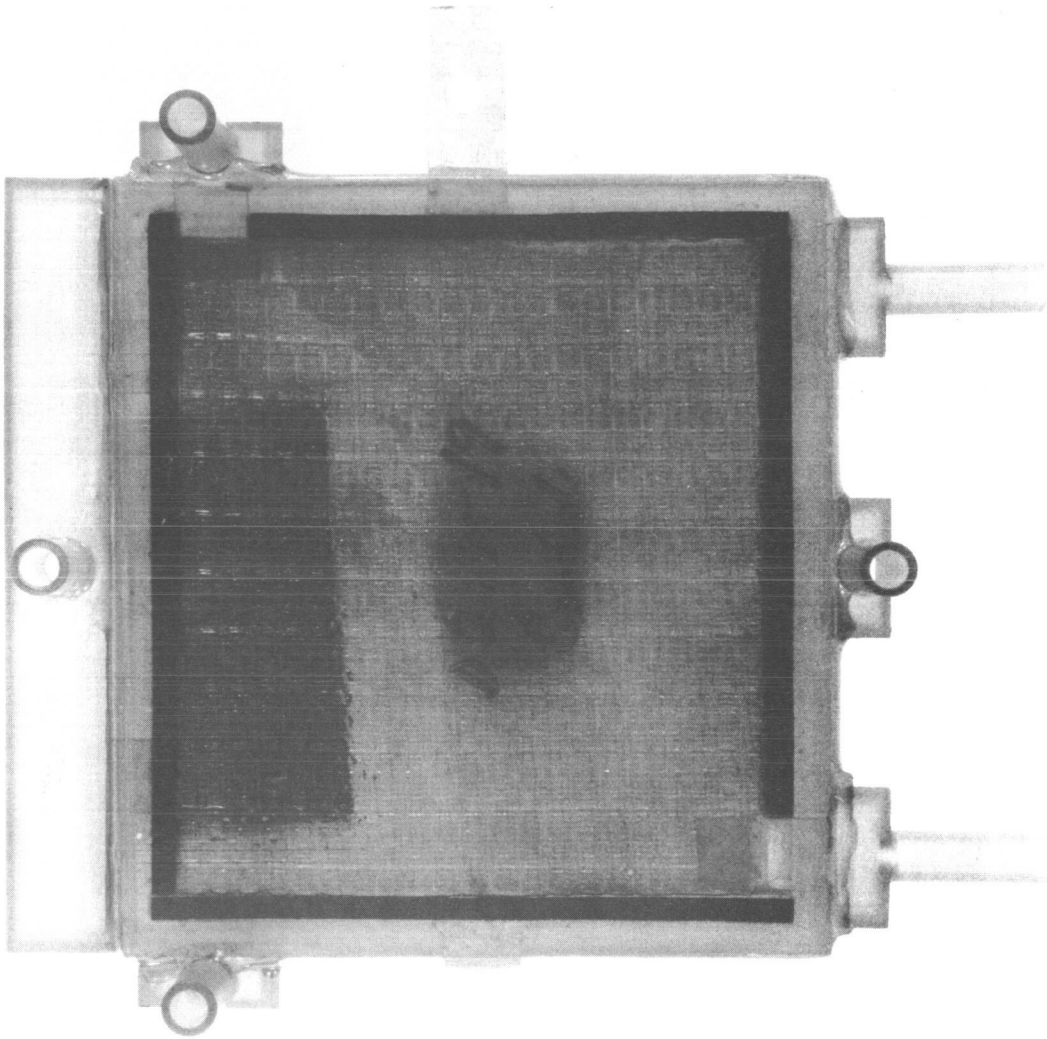
Fig. 6 - Small Stack for Operation at 15 psia, 1/8 Ft^2 Active Electrode Area.

$1\frac{1}{2}$

$1\frac{1}{8}$

$1\frac{1}{32}$

.025 THICK
(SILVER TAB)

$7\frac{1}{8}$

$\frac{3}{16}$ ENDPLATE

$\frac{3}{16}$ END PLATE
POLYSULFONE

.375 O.D. x .250 I.D.
(TYP.)

$5\frac{3}{8}$

KOH

$\frac{21}{32}$

H_2 IN

$\frac{3}{4}$

ELECTRODE
4.375 x 4.8
NET AREA =

$2\frac{7}{16}$

$4\frac{7}{8}$

CATHODE TAB

$\frac{4}{4}$
(TYP.)

$\frac{3}{16}$
(TYP.)

$1\frac{1}{2}$

O_2 OUT

$\frac{11}{16}$

KOH 1

.005 SILVER FRAME

.025 SILVER TAB

$\frac{3}{16}$

CLEAR EPOXY

SILVER EPOXY

ELECTRODE

ELECTRODE FRAMING
& TABBING DETAIL
NO SCALE

NOTE:
JIGS ARE USED TO PRECISELY
CONTROL EPOXY THICKNESS DURING
FRAMING & TABBING

END PLATE

SPACER GASKETS

END PLATE

LAY-U

NOTE

ALL

SEA

Fig. 7 - Schemat

FIND NO.	PART NO.	REQ'D	SHOWN ON DWG. NO.	DESCRIPTION

Page 17

$\frac{3}{8}$ (TYP)

OUT

O_2 IN

$\frac{3}{4}$

ALL FLUID CHANNELS FORMED WITH
.030 THICK x $\frac{1}{2}$ WIDE TEFLON STRIPS,
REMOVED AFTER PERIPHERAL EPOXY SETS.

:S
75
120 FT^2

1

$\frac{1}{2}$

$\frac{9}{32}$ DIA.

ANODE TAB

EPOXY

H_2 OUT

$\frac{11}{16}$

$\frac{1}{4}$ THICK EPOXY FILL BETWEEN END PLATES
ALL 4 SIDES

ALL FITTINGS MACHINED FROM
POLYSULFONE & EPOXIED IN PLACE

H_2 GAP SPACER MESH

ANODE

KOH GAP SPACER MESH

CATHODE

O_2 GAP SPACER MESH

REFERENCE DRAWINGS
350-021B-0
730-001A-0
730-002A-0

P PROCEDURE
O SCALE

APPROPRIATE PERIPHERAL AREAS
-ED WITH EPOXY DURING LAY-UP

c Drawing of Small Stack.

TABLE III

CONSTRUCTION FEATURES OF TEST STACKS

Stack No.	Anode	Cathode	Separators** Cathode	Separators** Electrolyte	Construction Completion Date
A-015	LAB-40nd*	LAB-40nd	S-1	S-2	Oct. '66
A-018	LAB-40nd	LAB-40nd	S-1	S-2	Oct. '66
A-021	LAB-40nd	LAB-40nd	S-1	S-2	Nov. '66
A-022	LAB-40nd	LAB-40nd	S-1	S-2	Nov. '66
A-035	UCC T-2	ChemCell H 9454N	S-1	S-2	Dec. '66
A-043	UCC T-2	LAB-40	S-1	S-2	Jan. '67
A-051	LAB-40 (A-II)	LAB-40 (A-II)	S-6	S-2	2/13/67
A-053	LAB-40 (A-II)	LAB-40 (A-II)	S-6	S-2	2/24/67
A-055	LAB-40 (A-II)	LAB-40 (A-II)	S-6	S-2	2/24/67
A-057	LAB-40 (B-II)	LAB-40 (B-II)	S-6	S-2	2/23/67
A-059	UCC T-2	LAB-40 (A-II)	S-6	S-2	2/20/67
A-060	UCC T-2	LAB-40 (A-II)	S-6	S-2	2/20/67
A-061	UCC T-2	LAB-40 (A-II)	S-6	S-2	2/20/67
A-066	UCC T-2	LAB-40 (A-II-2)	S-7	S-7	4/11/67
A-067	UCC T-2	LAB-40 (A-II-2)	S-7	S-7	4/17/67
A-068	LAB-40(A-II-2)	LAB-40(A-II-2)	S-7	S-7	4/14/67
A-069	UCC T-2	LAB-40(A-II-2)	S-7	S-7	4/19/67
A-070	UCC T-2	LAB-40(A-II-1)	S-7	S-7	4/19/67
A-071	UCC T-2	LAB-40(A-II-1)	S-7	S-7	4/11/67
A-072	UCC T-2	LAB-40(A-II-1)	S-7	S-7	4/14/67
A-073	UCC T-2	LAB-40(A-II-2)	S-7	S-7	4/19/67
A-074	UCC T-2	LAB-40(A-II-1)	S-7	S-7	4/17/67
A-075	LAB-40(A-II-1)	LAB-40(A-II-1)	S-7	S-7	5/03/67
A-077	UCC T-2	LAB-40(B-II-4)	S-7	S-7	4/11/67
A-078	UCC T-2	LAB-40(B-II-4)	S-7	S-7	4/17/67
A-079	LAB-40(B-II-4)	LAB-40(B-II-4)	S-7	S-7	4/14/67
A-081	UCC T-2	LAB-40 (143-A)	S-7	S-7	4/26/67
A-083	LAB-40(143-A)	LAB-40 (143-A)	S-7	S-7	5/10/67
A-084	UCC T-2	LAB-40 (143-A)	S-7	S-7	5/03/67
A-087	UCC T-2	LAB-40 (143-B)	S-7	S-7	4/26/67
A-089	LAB-40(143-B)	LAB-40 (143-B)	S-7	S-7	5/10/67
A-090	UCC T-2	LAB-40 (143-B)	S-7	S-7	5/13/67
A-201	LAB-40 (B-II)	LAB-40 (B-II)	S-7	S-7	4/10/67
A-202	UCC T-2	LAB-40 (A-II-2)	S-7	S-7	6/06/67
A-203	UCC T-2	LAB-40(A-II-2)	S-7	S-7	5/31/67
A-204	LAB-40 (A-II-2)	LAB-40(A-II-2)	S-7	S-7	6/09/67

*nd - No designation beyond LAB-40 is available for the earliest lots of American Cyanamid electrodes

** - Stack A-051 had S-6 separator in the anode space; all others had S-1 separators.

Identification of Separators:

S-1 Lamport style-7700 polypropylene; nominal thickness 0.093".
S-2 Exmet Code 10 expanded nylon 25-1 (stabilized).
S-6 Exmet expanded Teflon 25-1, flattened. (A single piece is flattened to 0.015"; 3 pieces were used in every instance and arranged so that the axis of the center piece was at right angles to the axes of the end pieces.)
S-7 Exmet expanded Teflon 25-1; heat treated but not flattened, 0.045" nominal thickness.

Similarly, autopsies showed that the standard polypropylene separator was not useful when exposed to O_2 at 90°C for long periods, and consequently both the electrolyte and cathode separators were selected from expanded-mesh Teflon materials. The S-1 Lamport polypropylene separator was satisfactory in the H_2 atmosphere of the anode compartment.

The electric current produced was collected by framing each of the electrodes in folded 0.005" silver foil, 3/16" wide after folding. The silver was sealed to the American Cyanamid Company's LAB-40 electrodes on the gas side with clear epoxy, and on the electrolyte side with silver epoxy. For the Union Carbide T-2 electrodes this arrangement was just reversed, so that the silver epoxy was on the nickel side of the anode. Furthermore, along one edge of each silver frame (usually on the gas side) was a 0.025" thick silver strip (3/16" wide) with an integrally welded tab to bring the current out of the cell. Tabs from anodes and cathodes were on opposite side of the cell.

The stack housing as a whole was made of polysulfone and epoxy. There were polysulfone end plates, with the electrodes and separating gaskets held between them by an epoxy bridge. The whole unit was sealed with Union Carbide Type-2774 epoxy with 2793 hardener in the ratio of 3:1, and oven cured at 50°C. The H_2 and O_2 inlets were at opposite top corners of the same face, the gas outlets at the diagonally opposite bottom corners. There were no orifices. Fluid slits 0.030" thick x 0.05" wide were provided as the electrolyte and oxygen inlets and outlets in the epoxy edge fill. Hydrogen slits were 0.040" thick to accommodate the thicker anode separator. The KOH inlet was at the bottom (center) with one inlet slit into the electrolyte space. At the top, two slits (one near each corner) opened into a plenum chamber which had one outlet in the center, with the top of this plenum being "gabled" to encourage egress of trapped gas. All the inlet and outlet tubes were polysulfone of 3/8" O.D., with 0.032" wall thickness.

The frame gaskets spacing the electrodes from each other and from the end plates were generally 3/16" wide, and of such thickness as to accommodate the separator properly. The gaskets were made from a special Neoprene rubber, treated with H_2SO_4, or from polysulfone. Both types were glued to the mating faces with epoxy in the cell subassembly. These gaskets and Teflon plugs (later pulled out to form the fluid passages) provided a seal during the pouring of the 1/4" thick epoxy in the four edges of each cell (successively).

The spacers in the cathode and electrolyte compartments are given in Table III. In the anode compartment, Type S-1 separators were used (Lamport Style-7700 polypropylene), except as noted in Table III.

3.2.3.2 Small Stacks for Use at 30 psia.

The construction of the pressure cells was identical with that of the regular stacks except for the addition of a reinforcing shell. This shell consisted of 1/4" Panelite end plates bolted together over the basic stack, and potted on all four sides with epoxy. A typical unit is shown in Fig. 8.

D-3394

Fig. 8 - Small Stack for Operation at 30 psia; Finished Cell is about 6-1/2 x 7".

3.2.3.3 Flight-Size Stacks.

The so-called flight-size stacks are basically scaled-up versions of the small stacks; they are not prototypes of the probable building blocks for the proposed 5-kw system. Front and rear views of the stack are shown in Fig. 9; details of construction are shown in Fig. 10; and a unit installed in a test stand is shown in Fig. 3; and in more detail in Fig. 11. The stacks were of Type-404, meaning there were four pairs of electrodes electrically parallel, as shown:

C = Cathode

A = Anode
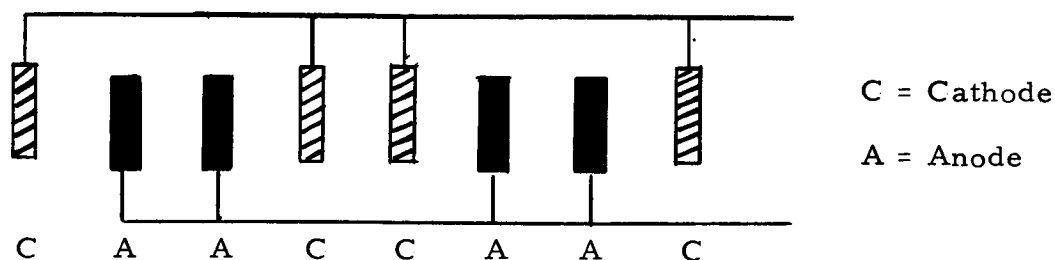
This modification was introduced for the usual reason: minimizing internal electrical resistance and current collection problems, and providing a safety margin in case one electrode should generate a substandard potential. Each electrode had an active area of 46.7 in^2, so that the cell had an active area of 1.3 ft^2; the contract called for a minimum of 1.0 ft^2.

Even though operation of these stacks at the specified 200 ASF required only 260 amperes, they are capable of handling 500 amperes. Sufficient conservatism was shown in the selection of materials, consideration of stresses, etc., that: a) all stacks could be operated at pressures even greater than 30 psia, and b) we were sure to test electrodes rather than stack construction.

The electrodes were framed in 0.005" silver foil, 1/4" wide on each side after folding. The silver was sealed to the American Cyanamid Company's LAB-40 electrodes on the gas side with clear epoxy, and on the electrolyte side with silver epoxy. For the Union Carbide Type-2 (T-2) electrodes this arrangement was again reversed, so that the silver epoxy was on the nickel (or gas) side of the anode. Furthermore, along one edge of each silver frame—usually on the gas side—was a 0.025" thick silver strip, 1/4" wide, with an integrally welded tab 1.5" wide to bring the current out of the cell.
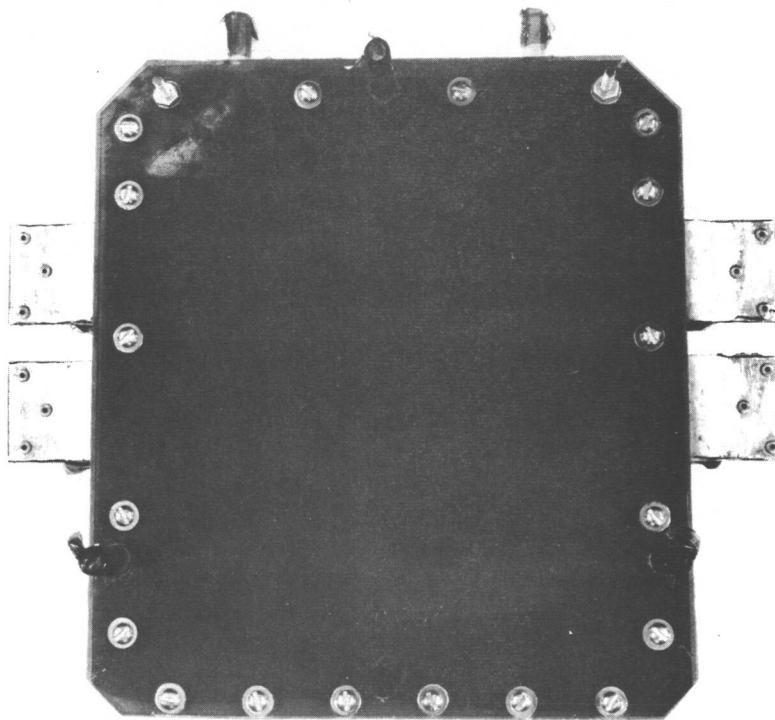
D-3403

Fig. 9a - Flight-Size Test Stack; Front View.
(Finished Stack is about 8-7/16" x 9-7/16".)
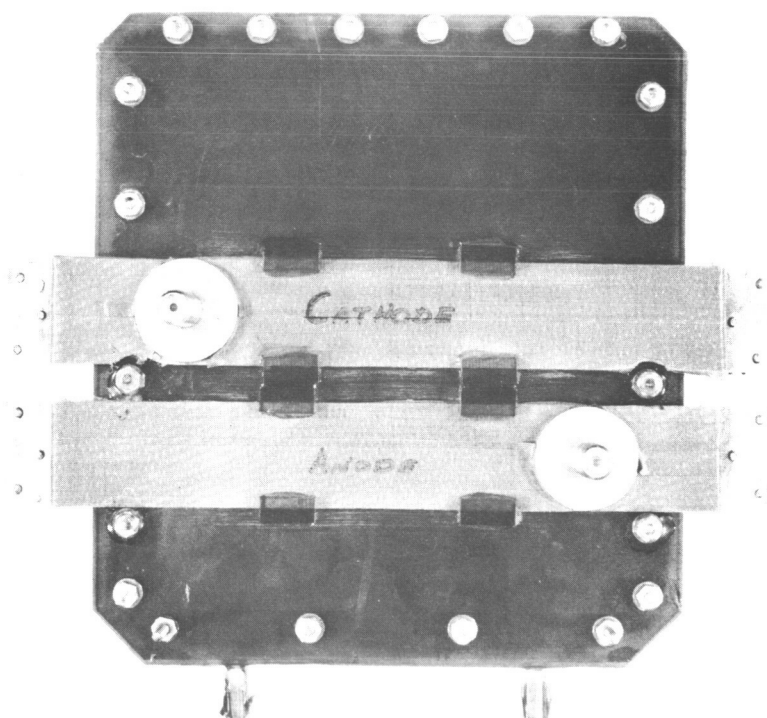
D-3402

Fig. 9b - Flight-Size Test Stack; Rear View Showing Bus Bars.

.375 O.D. × 250 I.D. (TYP)

KOH OUT

H₂ IN

ALL FITTING MACHINED FROM POLYSULFONE

+

−

$7\frac{7}{8}$

$3\frac{1}{32}$

H₂ IN

A A

FIRST EPOXY POUR (TYPICAL) $\frac{1}{8}$

O₂ OUT

KOH IN

O₂ OUT

$1\frac{11}{16}$

$4\frac{7}{16}$

O₂ OUT

O₂ SEPARATOR · KOH SEPARATOR · H₂ SEPARATOR · KOH SEPARATOR · O₂ SEPARATOR · KOH SEPARATOR · H₂ SEPARATOR · KOH SEPARATOR · O₂ SEPARATOR

NOZZLE END

END PLATE C-8 A-7 A-6 C-5 C-4 A-3 A-2 C-1 END PLATE

POLYSULFONE SPACER

LAY-UP PROCEDURE

SECOND EPOXY POUR POLYSULFONE ADDED TO LIMIT EPOXY RUNAWAY
FIRST EPOXY POUR

POLY (EPOX

ALUM

SOLDER ENDS TOGETHER

$\frac{1}{16}$ THK. OFHC COPPER

SILVER ELECTRODE TABS

SILVER SOLDER (TYP)

$\frac{1}{2}$-20 UNF-3 MAT'L.

SECTION A-A

NOTE:

ALL APPROPRIATE PERIPHERAL AREAS SEAL WITH EPOXY DURING LAY-UP. END PLATES HELD IN POSITION WITH UNIFORMLY APPLIED HYDRAULIC PRESSURE WHILE STUDS ARE TORQUED TO 15 IN# BY UNIFORM DISTRIBUTION OF BOLT LOADING.

REFERENCE DRAWINGS
800-029A-2
800-030A-6
800-031A-1
800-032A-1
800-045A-1

Fig. 10 - Schema

$8\frac{3}{4}$

KOH OUT

O_2 IN

$4\frac{5}{16}$

$9\frac{3}{8}$

$1\frac{1}{2}$ (TYP.)

+

−

B

B

KOH IN

H_2 OUT

KOH OUT

O_2 IN

+

−

KOH IN

H_2 OUT

DURING FIRST EPOXY POUR
ALL FLUID CHANNELS FORMED WITH
$5/8$ WIDE TEFLON STRIPS, REMOVED AFTER
PERIPHERAL EPOXY SETS.
.040 THICK FOR H_2 — .030 FOR O_2 & KOH

$-7\frac{7}{16}$

THESE 2 STUDS
2" LONG FOR
SUPPORT BRACKETS. EXTRA LENGTH
SPACED EQUALLY ON
BOTH SIDES.

SULFONE
ED TO ALUMINUM)

INUM

ECTRODE STACK

THD.
~ BRASS
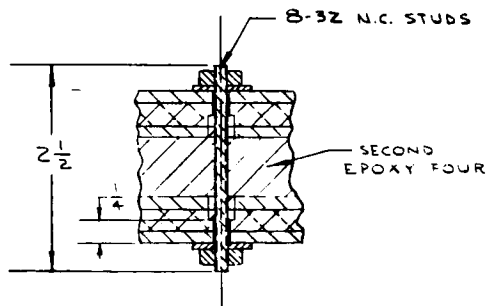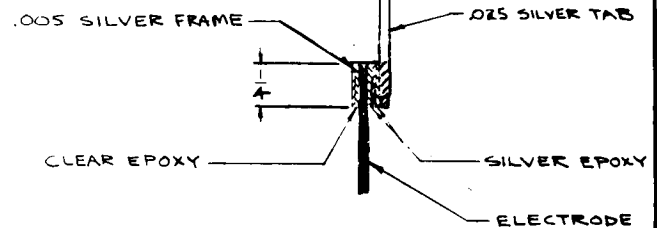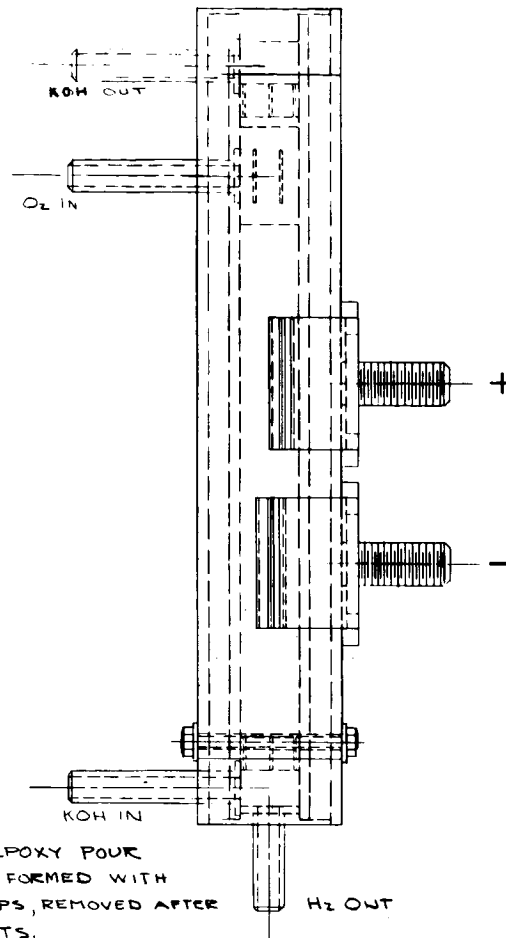
8-32 N.C. STUDS

$2\frac{1}{2}$

$\frac{1}{4}$

SECOND
EPOXY POUR

SECTION B-B

.005 SILVER FRAME

.025 SILVER TAB

$\frac{1}{4}$

CLEAR EPOXY

SILVER EPOXY

ELECTRODE

ELECTRODE FRAMING & TABBING DETAIL
NO SCALE

NOTE:
JIGS ARE USED TO PRECISELY
CONTROL EPOXY THICKNESS
DURING FRAMING & TABBING

ELECTRODES $7\frac{3}{16}$ x $7\frac{7}{8}$
≈ 46.7 SQ. IN. (NET) ELECTRODE AREA
1.3 SQ. FT. STACK (404 CONN.)

ic Drawing of Flight-Size Stack.

D-3438

Fig. 11b - Rear View of Flight-Size Stack in Test Stand C-3.

D-3436

Fig. 11a - Front View of Flight-Size Stack in Test Stand C-10, Showing Panel and Electrolyte Tank.

Tabs, later to be paralleled, were brought out on opposite sides in deference to the matching up of edge spacer thickness with fluid gap mesh thickness. The tabs were joined externally to appropriate copper bus bars, as shown in Fig. 9b. There were also peripheral spacing strips of 3/16"-wide polysulfone. This design does not incorporate fluid orifices; slits with the dimensions shown in Fig. 10 served as gas and electrolyte passages.

The end plates consisted of 1/4" aluminum sheets sandwiched between two 1/8" polysulfone sheets, glued together with epoxy. After the stack was aligned and assembled in a jig, with epoxy applied to all the appropriate mating surfaces, the stack was compressed slowly and uniformly with hydraulic pressure to assure uniform and continuous bonding by the adhesive. While the pressure was on the stack, bolts were inserted and tightened following a uniform torquing scheme, up to an applied torque of 15 inch-pounds per bolt. After bolting, the sides of the stack were potted with 2774/2793 epoxy. All nozzles were polysulfone.

3.2.4 Tests at 200 ASF.

3.2.4.1 Introduction and Criteria.

One objective of this work was the development of a fuel cell system using the Union Carbide concept of fuel cell operation, which will meet the following requirements:

a) an initial potential of 0.90 volt at 200 ASF;

b) a rate of voltage degradation not exceeding 0.020 volt/500 hours. Data to be shown below will indicate that at 15 psia criterion "a)", even though it was met by ten of the fifteen small stacks tested, is not relatable to long-term stack performance. Requirement a) was met by all stacks tested at 30 psia. The situation with regard to requirement "b)" will be discussed below; since several cells are still on test, the final tabulation cannot yet be compiled.

The contract's Task 2 involves the preliminary design of a 5-kw system with a life of at least 3000 hours. Combination of the aforementioned voltage requirements indicates that the system would show at least 0.78 volt at the end of life, and hence 0.78 was adopted as the cut-off voltage for the life tests. The results of the tests will be discussed by certain groupings. The figures pertaining to the individual cells will be found in Section 3.2.6. These figures are arranged in three groups: Figures 18 through 50 give the history

of each cell in the form of a time-versus-voltage plot, with the KOH normality plotted on the same graph. Figures 51 through 83 show the rate of electrolyte leakage through the anodes or cathodes in milliliters of electrolyte/hour· ft² of electrode surface. Figures 84 through 119 are the polarization curves. Usually there is just one graph per cell showing an early or initial polarization curve, one taken five hundred hours later, and one taken near the end of life. In a few cases where some special procedure was involved a second graph shows the polarization curves taken shortly before and after the operation.

3.2.4.2 Small Stacks Run at 15 psia.

The results of this initial series of tests are very encouraging. They are summarized in Table IV, from which it may be seen that requirement "a)" was met by ten of the fifteen stacks tested, and five of these ten also met requirement "b)". (Following standard rounding-off procedures, 0.895 becomes 0.90 volt.) Furthermore, three of the five cells which did not meet "a)" showed the required low voltage degradation. The two best stacks showed lives of 2,140 and 2,160 hours, but only the first of them would have been acceptable under criterion "a)". Five stacks lived longer than 1500 hours, but only two of these had initial potentials of 0.90 v. These data suggest that, at 200 ASF and 15 psia, the stacks with a lower initial potential are likely to have longer lives and less voltage degradation.

Eight of the fifteen cells discussed here had been made with Union Carbide Corporation's Type-2 anodes. Of these, 5 had lives in excess of 1000 hours. Similarly four cells with LAB-40 anodes had lifetimes of greater than 1000 hours. Both types of cells also showed very similar leakage behavior. On these bases, no distinction can be drawn at this time between LAB-40 and T-2 anodes. But there are distinctions between the various lots of LAB-40 electrodes. These are discussed in Section 3.2.4.5. The marked similarity of the cells is also illustrated in their polarization curves. Figures 12 and 13 show that all but two of the polarization curves taken initially (Fig. 12) and some 500 hours later (Fig. 13) fall within a well-defined region, even though the width of the band increases with age. The two exceptions, Stacks A-081 and A-087, did not perform well. It should also be noted that four tests were terminated by problems of a mechanical nature rather than electrochemical degradation.

TABLE IV

PERFORMANCE SUMMARY, SMALL STACKS AT 200 ASF, 15 PSIA

Cell No.	Startup Date	Peak	Cell Voltage at 100 Hrs.	300 Hrs.	500 Hrs.	700 Hrs.	900 Hrs.	1000 Hrs.	1500 Hrs.	End*	Lifetime Hrs.	KOH Flow, ml/min at ΔP at 1.5" KOH at 90°C
A015	12/01/66	.880	.880	.880	.880	.880	.875	.870	.790	----	1620	205
A021	1/09/67	.880	.880	.870	.850	.835	.825	.810	.800	----	1910	170
A022	1/04/67	.885	.880	----	----	----	----	----	----	----	199	100
	2/03/67°	----	----	.880	.870	.860	.850	.850	----	.830**	1394	
A043	1/19/67	.910	.900	.900	.905	.900	.890	----	----	.875***	918	80
A055	3/10/67	.905	.905	.890	.890	.870	.870	.845	.835	----	2140	80
A059	2/22/67	.870	.870	.870	.870	.870	.870	.870	.850	----	2160	210
A060	2/27/67	.900	.900	.880	.875	.860	.855	.860	----	----	1370	140
A066	4/12/67	.910	.910	.910	.905	.885	.870	.870	----	.820#	1386	80
A068	4/18/67	.910	.905	.890	.865	.815	.790	----	----	----	930	20
A071	4/14/67	.890	.890	.890	.890	.870	.840	----	----	----	945	70
A075	5/09/67	.910	.890	.890	.870	.825	.780	----	----	----	950	--
A077	4/14/67	.900	.900	.915	.905	.885	.885	.890	----	.890##	1030	65
A079	4/17/67	.895	.895	.890	.875	.865	.845	.840	.810	----	1580	--
A081	4/28/67	.905	.900	.885	(.780)	----	----	----	----	----	690	120
A087	4/27/67	.900	.895	.880	.840	.820	----	----	----	----	710	78

* End of life is defined as the time when the cell voltage falls below 0.780 v, unless some higher voltage is shown.

** Shut down because of internal mechanical damage.

*** Shut down because of apparent KOH blockage.

Failed to run properly after a temporary H_2 outage.

Shut down because the polysulfone case split.

° Stack washed and dried, and returned to test. Time runs continuously.
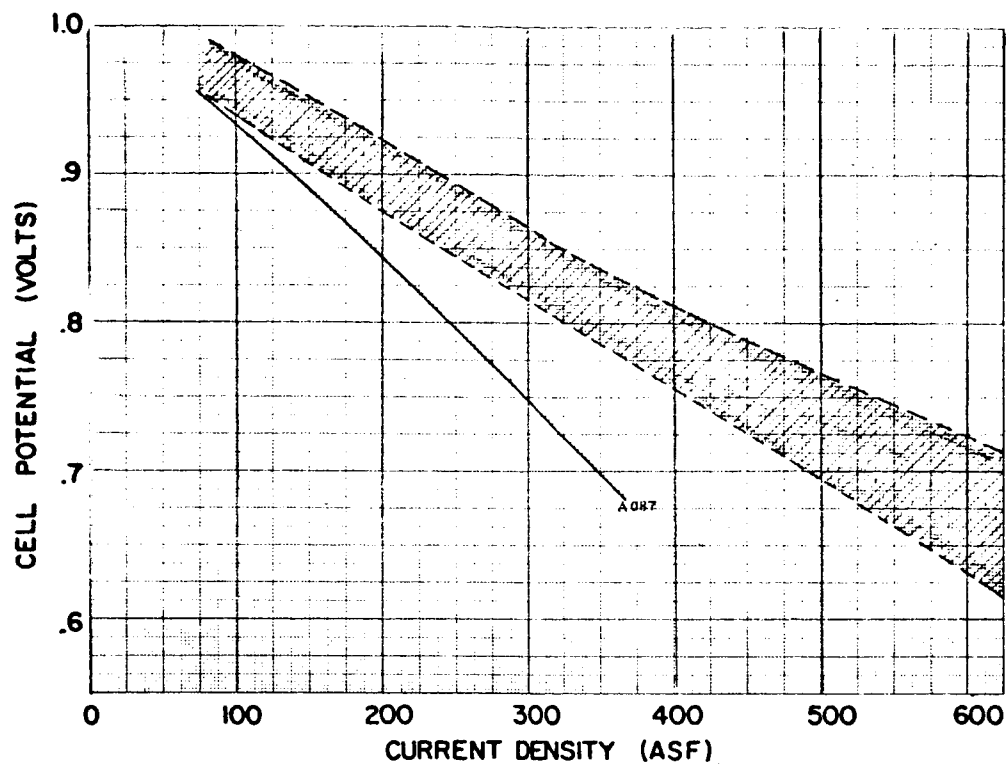
D-3464

Fig. 12 - Envelope of Initial or Early Polarization Curves of Small Stacks.
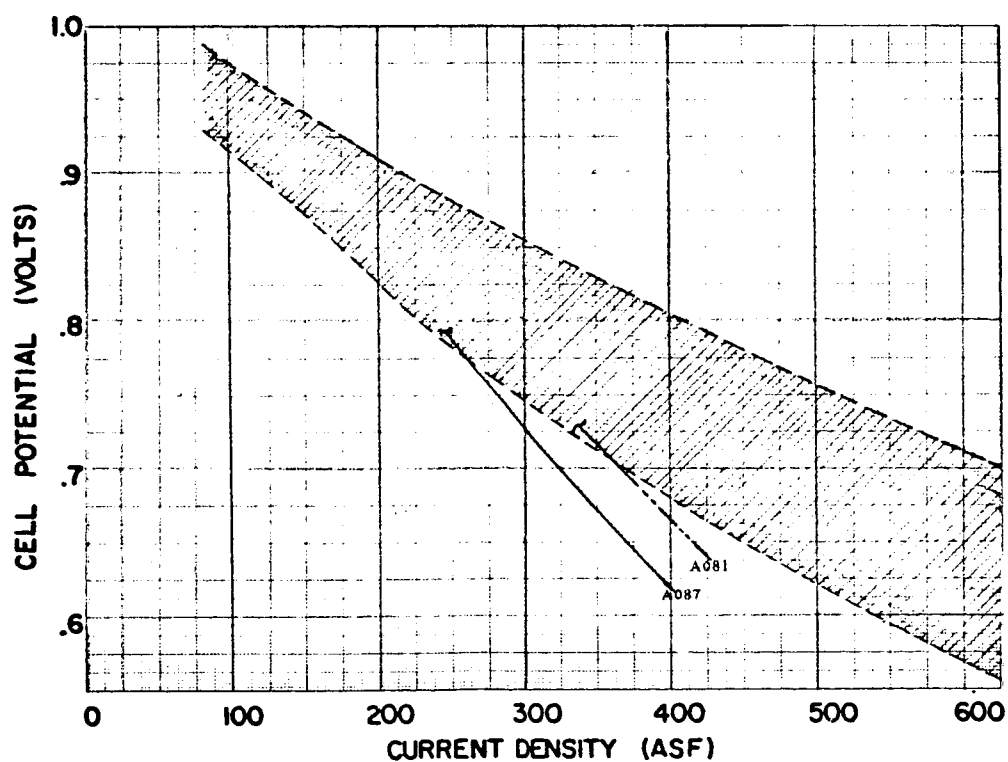
D-3465

Fig. 13 - Envelope of Polarization Curves of Small Stacks,
500 Hours Later than Fig. 12.

Stack A-015 was the first unit to be put on test in the stack test facility and also one of the best units tested, in spite of the fact that the test stand was also being evaluated in this first run. The cell's time-voltage history is given in Fig. 18, its leakage in Fig. 51, and polarization curves in Figs. 84 and 85. (The IR-free curves are marked by the letter "F" to distinguish them from the IR-included data.) A-015 maintained its initial 0.88 volt potential for some 750 hours. Then it began to drop off, reached 0.78 v at 1,620 hours, and was removed from test at 1,630 hours at 0.75 v to be washed and dried; however, the polarization curve showed very little improvement. It was returned to test at only 0.78 v, and showed 0.74 v when it was again removed at 1,797 hours. Test conditions deviated from the norm in that the KOH flow was high, being around 360 ml/min. at a 5" ΔP. This was due to awkwardness in adjusting flow conditions on this first test stand.

One item of considerable interest is the internal resistance of the unit. Since certain fuel cell designs seem to suffer from a gradual increase in internal resistance, we calculated this parameter for A-015 from interrupter readings taken over the life of the stack. The internal resistivity, defined as (IR-free minus IR-included)/current density, was 0.3 milliohm· ft² throughout the life of the unit, as computed from nine different sets of data. The time-independence of the low resistivity of this type of fuel cell stack was confirmed on numerous other stacks in this series.

Autopsy of the unit after failure showed: a) the Exmet nylon KOH separator had largely disintegrated; b) both electrodes were rather wet on the back. On the anode about two-thirds of the surface was wet and the Teflon adhesion was fair. The constant, low resistivity is all the more remarkable in the light of this finding.

With Stack A-021 (Figs. 20 53, and 87) the main problem was maintaining the right temperature. This stack suffered four slight temperature excursions up to 100°C, one each near 100 and 200 hours, respectively, and two at about 825 hours. It also experienced two involuntary shutdowns; one due to a main (CEI) power failure during the early morning hours on February 16, 1967, corresponding to a stack age of about 900 hours, and one of shorter duration at 1,340 hours due to an H_2 house supply failure; none of these events seem to have had a serious effect on the unit. It dropped below 0.78 v after 1,910 hours on load, and was removed from test because of a KOH-pump failure at 2,030 hours (0.695 v). For some unknown reason the pressure differential across the oxygen cavity rose gradually from about 0.4" w.c. at 150 hours to 4" at

740 hours, and then suddenly dropped again to the normal range for a few hundred hours. By 1800 hours, it had risen again all the way to 10", and at that point the manometer had to be clamped off. During this second period of high pressure drop the cell potential was quite sensitive to the O_2 flow rate. Experiments on the cold cell after shutdown did not provide any insight into the cause of this observation.

Stack A-022 (Figs. 21, 54, and 89) has a peculiar history, and was probably somehow defective from the beginning. At first it maintained its initial voltage, but at about 170 hours it began to drop off quite rapidly, and at 199 hours it had fallen to 0.78 v. We then reduced the load to 100 ASF and the voltage rose to 0.905 v; the unit was purged with argon, then the gas connections were reversed subsequently so that the anode became cathode, and vice versa. This yielded no benefits; the voltage again dropped to 0.675 v at 221 hours, and then suddenly reversed. At that time the unit was removed, washed, and dried. Upon removing the Styrofoam insulation one could see that the cathode separator had turned dark red and had disintegrated. Most of it washed out during the washing step. Nonetheless, the unit was returned to test; it behaved admirably.

In its second lease on life, A-022 ran for an additional 1,173 hours from 0.880 to 0.830 v, but then it suddenly failed with violent blowing through of gas and a very low potential, even on open circuit. This suggested an internal short. Autopsy revealed that there had been some strong local overheating. Two holes were burned completely through the polysulfone end plates; one on the anode, the other on the cathode side, just directly opposite each other. The nylon KOH separator was intact except in the vicinity of the burned areas where it had disappeared. As indicated above, the Lamport polypropylene spacer in the cathode compartment had disappeared completely. The Teflon cathode backing was wet over much of its surface. Adhesion of the Teflon was only slight, but the back of the cathode (under the Teflon) was only partially wet. The Lamport spacer in the anode compartment was intact except in the vicinity of the burnt area. There was no KOH in the gas space, and the back of the Teflon was almost completely dry, but the Teflon adhesion was quite poor, and about two-thirds of the anode under the Teflon was wet.

This stack also suffered from some interruptions of operations. There was an involuntary shutdown due to the power failure on February 16, 1967, at 524 hours, another brief interruption on March 6, 1967 at 960 hours, and a deliberate shutdown at 984 hours to replace a noisy KOH pump.

Stack A-043 (hybrid, Figs. 23, 56, and 92) was also a victim of test-stand problems. It had an initial voltage of 0.900 v, rose briefly to 0.910 at about 250 hours, and remained at 0.900 v to over 700 hours. It suffered accidental shutdowns due to the general power failure at 652 hours and due to low KOH level in the system because of leaks through cracked nylon fittings at 207 hours, 722 hours, and finally 918 hours. After the last shutdown, we were unable to restart the unit. After the system was repaired, the stack showed a low potential which decreased with increasing H_2 pressure. Furthermore, KOH flow was very low and remained so even when the KOH inlet pressure was raised to 30". Back-flushing was impossible. The nylon KOH separator had almost completely dis-integrated. It is suspected that blockage of the KOH-outlet port was the cause of cell overheating which, in turn, damaged the electrodes. Incidentally, this unit had supported 650 ASF in a polarization test.

Stack A-055 (Figs. 26, 59, and 95) was one of the best stacks tested. The initial potential of 0.905 v decayed only slowly for over a thousand hours, and then more rapidly so that it crossed the 0.78 v mark at 1,270 hours. The unit was shut down (i. e. , heat and electrolyte circulation shut off, H_2 replaced with Ar) at 1,359 hours and 0.730 v, but then reactivated (by returning H_2, electrolyte circulation, and heat) for further tests. Upon reactivation the unit showed 0.910 v but dropped steeply to 0.770 v at 1,520 hours; at this point dilute gas tests* (show-ing the anode in trouble) apparently rejuvenated the unit to 0.835 v and resulted in a much slower decay rate, so that it reached cut-off point again at only 2,070 hours. Another deactivation, reactivation, and dilute gas test (still only the anode in trouble) did not give the improvement found earlier, and the unit was finally taken off test at 0.765 v after 2,219 hours under load. Operating life has been recorded at 2,140 hours, since nothing remedial was done to the unit except turning it on and off at the various earlier times. This stack, too, suffered a few involuntary shutdowns which, however, seemed to have no particularly adverse effects.

---

* Dilute gas tests are a diagnostic tool in which the stacks are run either with scrubbed air instead of O_2, or with a 15% H_2-85% Ar mixture instead of the 100% H_2. A small potential drop is predicted for good electrodes, with a steady-state voltage reached quickly. If either gas distribution or diffusion are impeded, inerts build up rapidly, a much lower potential is reached ultimately, and the time required to reach same is much longer. In extreme cases the steady state is never reached. Return to pure H_2 or O_2 generally brings the stack back to the earlier potential, and sometimes has a very interesting, unexpected rejuven-ating effect, i. e. , the stack returns to a higher potential than it showed before the dilute gas test.

Stack A-059 (hybrid, Figs. 27, 60, and 96) maintained its initial potential of 0.870 v for some 1300 hours before beginning to decline. At 2,214 hours, 0.680 v, the load was reduced to 100 ASF, the voltage returned to 0.835, where it remained for the rest of the test which was concluded at 2,284 hours to make the test position available for another stack. Dilute gas tests at 1,968 and 2,209 hours both agree and indicate decaying anodes, and this is confirmed by measurements with a Zn-reference electrode. The stack showed about a 20% decrease in KOH flow during its life from the initial value, and at fixed ΔP. It was shut down deliberately at 1,458 hours to permit repair of a leak in the KOH system. For comparison purposes, the time-voltage plot (Fig. 27) for this stack includes values of the potential at 400 ASF. These were taken from the weekly polarization curves.

Stack A-060 (hybrid, Figs. 28, 61, and 97) was a companion test to A-059 but did not perform quite as well, dropping from 0.900 to 0.780 v in 1,370 hours, and to 0.750 v in 1,514 hours. Dilute gas tests at 1,420 and 1,495 hours indicated that both electrodes had decayed, but the anodes more than the cathodes. After both tests the potential was somewhat higher than it had been before the tests, but we were unable to duplicate this effect with sudden surges or increased pressures or flow rates of the usual reactant gases.

Stack A-066 (hybrid, Figs. 30, 63, and 99) ran quite well (dropped from 0.91 to 0.82 v in almost 1400 hours) until a failure in the house H_2 supply shut the unit down late one afternoon. While it apparently restarted after the shutdown, it was found next morning to show only 0.625 v. at 120 ASF. Reducing the load to 50 ASF gave a potential of 0.930, which dropped to 0.895 v in 55 hours. The load was then increased to 100 ASF, at which time it dropped from 0.825 vo 0.790 v in 66 hours. The test was terminated at 1,522 hours. Gas pressures had to be reduced at 1,425 hours to avoid blowthrough. Dilute gas tests at 1,453 hours showed both electrodes had decayed, but the anode more so than the cathode.

Stack A-068 (Figs. 31, 64, and 100) was in some way defective. The gas flows were normal, but the KOH flow was very low, and a ΔP of 10" and even more was required to maintain adequate flow and to keep the cell temperature up. The cell dropped from 0.910 v to the cut-off point in 930 hours. The voltage dropped to 0.76 in 836 hours; dilute gas tests were performed (showing anode problems) which returned the voltage to 0.80 v with the cutoff reached

the second time at 930 hours. At 978 hours, 0.75 v, the load was reduced to 50 ASF, giving 0.96 v, but that potential had decayed to 0.905 v at 1,054 hours when the test was terminated.

This stack was also utilized for an experiment which must be deemed inconclusive because of the other problems encountered. Starting at 506 hours the O_2 gas was humidified but the change in conditions did not affect the downward slope of the voltage curve in any way.

Stack A-071 (Figs. 33, 66, and 102) showed substantially no electrolyte leakage, but yet did not perform well. This inverse relation between two desirable attributes has been observed frequently. The stack held an initial potential of 0.89 v for some 300+ hours, then briefly rose to an even higher potential of 0.905 v and thereafter began to decline. It still showed 0.87 v at 920 hours, dropped to 0.82 v in the next 144 hours, and then to 0.70 v in two more days. Dilute gas tests and measurements with reference electrodes both showed the anode to be slightly weaker than the cathode.

Stack A-075 (Figs. 37, 70, and 107) suffered two involuntary shut-downs during its life. At about 0300 hours on June 7, 1967, there was a general power failure at 687 hours of stack life, and at 748 hours there was a brief interruption of the house H_2 supply. Stack performance thereafter fell off quite rapidly, but the dilute gas tests at 912 hours (anodes fairly poor) gave it new vitality, so that it managed to reach 950 hours. But once it began to fail it failed rapidly so that even at 100 ASF it did not last, and it was removed from test at 980 hours.

Stack A-077 (Figs. 38, 71, and 108) operated at excellent potentials until prematurely terminated by mechanical problems. It started at 0.900 v while the KOH concentration was somewhat low (12-13 N), but by about 310 hours when the KOH concentration had passed the 14 N mark the voltage had climbed to 0.915, where it remained for about six days; it then dropped slowly to 0.890 v just beyond the 1000-hour mark. At 1,011 hours a humidifier was installed in the O_2 inlet immediately before the cell port. Either the installation was carried out improperly or a strain was placed on the stack port; in any case, the next morning the KOH discharge manifold was found cracked and leaking badly. There were also problems with the O_2 plumbing; during the period of this trouble, the ethylene glycol manometer fluid had somehow been sucked into the cathode compartment and had wetted the electrode. The stack was removed, cleaned with isopropanol (to remove the ethylene glycol), repaired and returned to test, but

failed to come up to proper potential under load. Both voltage decay and the polarization curves indicate that this was one of the best stacks tested.

Stack A-079 (Figs. 40, 73, and 110) was another instance where the dilute gas tests apparently had a life-prolonging effect, the nature of which is not at all understood. A-079 started at 0.90 v, declined uniformly to reach 0.80 v in about 1,250 hours, and then dropped off more rapidly. Dilute gas tests at 1,395 hours brought the voltage back up to 0.84 v but not for long; the unit went below the cut-off point at 1,580 hours and was removed from test at 0.72 v after 1,683 hours. The dilute gas tests had shown a rather poor anode, but a cathode in good shape.

Stack A-081 (Figs. 41, 74, and 111) was another instance of a poor stack which, however, showed no leakage through the electrodes. Some of the early voltage readings are unreliable because of a poor electrical connection, but even after that was corrected the voltage had decayed from the initial 0.905 v to 0.78 v in about 500 hours. Performing the dilute gas tests (both anode and cathode were very poor) boosted the potential back to 0.84 v. Unfortunately, it again decayed rapidly and reached the nominal cutoff at 690 hours. At 748 hours (0.700 v), the current was reduced to 50 ASF, and the potential rose to 0.830 v. It decayed to 0.805 v by 812 hours and the unit was removed from test.

Stack A-087 (hybrid, Figs. 44, 77, and 113) was also a poor stack; here the matter is clouded by the development of a blockage in the KOH portion of the stack at some time. The stack started at 0.900 v and then declined uniformly to 0.820 v at about 700 hours. Thereafter, it dropped quickly to 0.690 v at 741 hours, at which point the load was reduced to 100 ASF. The new potential of 0.845 v also dropped fairly rapidly, and at 811 hours the load was again reduced, this time to 50 ASF, giving 0.890 v. At about that time, reduced KOH flow became noticeable, so the unit was removed for examination, probing, and forward and reverse flushing. No obstructions or foreign material were found, hence the stack was returned to test, but continued to perform poorly, and was removed finally at 907 hours. Dilute gas tests at 674 and 860 hours had showed a fair cathode but a poor anode, and the anode seemed to have decayed further between the tests.

This stack is one of two units whose polarization curves fall below the envelope of the other 13 stacks, as evident in Figs. 12 and 13. All these defects are probably related.

As can be seen from Fig. 42, the KOH outlet temperature varied considerably during part of this test. The temperature remained near 90°C through the 30th day (720 hours). Then it fell rapidly, reaching room temperature by the 34th day. The KOH flow through the cell could not be increased sufficiently, so the cell was back-flushed in place. Thereafter the temperature could be raised to 63°C the next day. The stack was then removed from test at 835 hours for visual examination and probing. No obstruction could be found. Back-flushing showed easy flow. The KOH solution used for flushing showed no trace of solids. The stack was equipped with a new KOH inlet fitting and returned to test. A KOH flow test showed 56 ml/min. flowing at 1.5" KOH ΔP, but the temperature still could not be returned to 90°C.

After the operation, the electrical performance remained poor. Probably the already-weak anode was damaged even further by operation at the high current densities and low temperatures. Both Stacks A-081 and A-087 showed poor performance, as reflected in their time-voltage and polarization curves. Stack A-081 is not included in the envelope of initial polarization curves (Fig. 12) since we suspected the data might be unreliable because of a loose connection. Had it been included, it would have fallen below A-087. The defect had been remedied by the time the curves in the second envelope (Fig. 13) were taken. In Fig. 13, only A-087 is poorer than A-081. Probably the blame can be ascribed to the LAB-40 (143-A & 143-B) electrodes.

At the time the first four cells in this group (A-015, A-021, A-022, and A-043) were tested, the dilute-gas test had not yet been developed, and consequently evidence is not available as to whether the anode or cathode showed greater degradation in potential. However, in the other 11 cells, the preponderance of evidence points toward the anode as being the weaker of the two electrodes. This generalization (which applies to both T-2 and LAB-40 anodes) must be somewhat tempered by the fact that a shutdown in the H_2 supply occurred while several of these cells were on test. This may have biased some of the results.

3.2.4.3 Small Stacks Run at 30 psia.

There is no a priori reason why any device intended for use elsewhere than the surface of the earth should run at a pressure of 15 psia rather than some other convenient or advantageous pressure. There are sound reasons (e.g., the change in chemical activity with pressure) for expecting higher poten-

tials from a fuel cell at higher pressures, and there may be other advantages Therefore some units were tested at 30 psia. The results are summarized in Table V.

Seven stacks were built for this portion of the program. Two units were never tested because of mechanical problems, one is still waiting to go on test. The four stacks which actually started all showed initial potentials of 0.920 v, and the two longest-running stacks (which are still on test) show voltage degradations of only 0.025 and 0.030 v for the initial 1000 hours. One test has had to be interrupted because of a KOH leakage problem in the pressurized system, one test had to be concluded because of test system malfunctioning.

As indicated, the first two units put on test both failed because of mechanical problems or damage to the electrodes. Stack A-057 was used to check out the new pressure test stand design, and appeared to operate properly at 15 psia, showing 0.890 v at 200 ASF and 77°C; however, when we started to pressurize the system, the stack suddenly caught fire, and was sufficiently damaged to forestall a meaningful autopsy. The suspicion was that the pressure did not build up uniformly but, rather, the buildup in the electrolyte portion lagged; thus, the ΔP across the electrodes became large enough to rupture one or both of the electrodes and bring the reactant gases in contact. Even if the electrodes did not actually rupture, there was probably sufficient blow-through to lead to the formation of a combustible mixture.

Stack A-067 never went on test. At activation, copious H_2 blow-through in the KOH was observed which did not stop even after the unit sat active but on open circuit overnight. If there was any O_2 blow-through, it was masked by the H_2. On the other hand, during this first night while on open circuit, a cathode leak rate of 9.4 ml KOH/hr·ft^2 was observed. Since the blow-through had not abated, the unit was removed from test. It might have been damaged during startup because a very unorthodox procedure was followed: Hot KOH (perhaps 95°C) was admitted to the cold stack, and the resulting thermal stresses might have damaged the cell.

Stack A-070 is still waiting to go on test.

Stack A-074 (Figs. 36, 69, 105, and 106) showed excellent electrical behavior, dropping only 0.025 v in 1000 hours; however, its leak rate became very high so that at 1,152 hours it was depressurized, changed to an atmospheric pressure test stand and returned to test at 15 psia. It is still doing well, showing

TABLE V

PERFORMANCE SUMMARY, 1/8 FT² STACKS AT 30 PSIA

Stack No.	Starting Date 1967	Time of Pressurization (Hrs)	Voltage at									As of 0900, 1 July 1967		Lifetime* Hrs
			Peak	100 Hrs	300 Hrs	500 Hrs	700 Hrs	900 Hrs	1000 Hrs	1500 Hrs	End*	V	Hrs	
A-057	4/05	28	Failed mechanically on pressurization.											
A-067	4/19	Removed because of mechanical defect—high KOH leakage through electrodes.												
A-070	Not yet on test.													
A-074	4/19	29	.920	.920	.910	.900	.890	.890	.895	.840**	--	.840	1745	
A-078	4/21	76	.920	.920	.905	.910	.905	.895	.890	.895	--	.880	1697	
A-083	6/07	50	.920	.920	--						.900	--	--	180
A-089	6/23	74	.920	.920	Test interrupted at 142 hrs because of KOH leak. Still active.									

*   Lifetime is defined as the time on test before the potential falls below 0.78 volt, unless there is an entry in the End-of-Life column.

**   At 15 psia. Stack was removed from 30 psia test stand and put on test at 15 psia because of excessive leakage at the higher pressure.

0. 84 v at 1,745 hours, and the leakage has decreased significantly. The first polarization curve shows the increase in performance upon pressurization, and the second one the decrease upon reduction of pressure.

Stack A-078 (Figs. 39, 72, and 109) still on test and performing well, dropped from an initial 0.920 v to 0.895 v at 1,697 hours; it leaks slightly through both electrodes.

Stack A-083 (Figs. 42 and 75) suffered several system operation mishaps which apparently led to its early demise. At 285 hours it was shut down, showing 0.810 v at 50 ASF.

Stack A-089 (Figs. 47, 78, and 114) started off excellently at 0.92 v; the test was interrupted at 170 hours to permit solving a test stand problem. Thereafter, the test will be resumed.

3. 2. 4. 4 Flight-Size Stacks.

Four of the larger units were also placed on test, and the results are summarized in Table VI. The two stacks operating at 30 psia would have met criterion "a)"—the initial voltage requirement of 0.90 v—but the two 15-psia stacks would not. All four stacks met criterion "b)"—the low rate of voltage degradation. Three of the stacks are still on test.

Stack A-201 (Figs. 47, 80, and 116), the fourth stack, was used to check out the new large-pressure test stand. It performed well, showing 0.880 v at 824 hours—down only 0.020 v from its peak of 0.900 v. It then began to drop fairly rapidly, with the next few voltage readings taken one day apart being 0.875, 0.860, 0.825, 0.845 (due to increased O_2 pressure), and 0.795 at 974 hours. Increasing the oxygen pressure had restored the voltage to higher values, but caused unacceptably high blowing-through. The unit was therefore shut down for an immediate autopsy. This autopsy, however, revealed no basic flaw. All the edge seals were intact. A small piece of the active area on one cathode was missing but was probably removed during the disassembly. The separators had retained their dimensions and had not disintegrated, nor did they show any signs of attack. Adhesion of the Teflon to the active layers on two of the cathodes was poor, the Teflon was easily removed, and the electrodes were wet underneath. Bonding of Teflon to the anode active layers—though not strong—was real, and the anodes were dry.

TABLE VI

PERFORMANCE SUMMARY FOR FLIGHT SIZE STACKS

| Stack No. | Starting Date 1967 | Voltage at | | | | | | | Lifetime Hrs | As of 0900 1 July 1967 | | KOH Flow ml/min at 1.5" ΔP |
		Peak	100 Hrs	300 Hrs	500 Hrs	700 Hrs	900 Hrs	End of Life		V	Hrs	
A-201**	4/11	.900	.900	.900	.885	.880	.825	.795	974			
A-202*	6/13	.880	.880	.880	.880					.880	426	255
A-203**	6/06	.910	.910	.905	.915***					.910	584	260
A-204*	6/15	.865	.865	.865						.865	374	245

*   At 15 psia.

** At 30 psia.

*** Potential rose 0.020 volt after shutdown at 369 hours to make changes in house O_2 supply.

The stack had started to leak slightly through the anodes at about 500 hours, markedly through the cathodes at about 300 hours. Leakage decreased when the O_2 pressure was increased but, as mentioned above, the higher pressure caused excessive blowing-through.

The three stacks still on test are performing well. Fig. 118, the polarization curves for Stack A-203, shows the improved performance achieved on pressurization. Fig. 119 shows two polarization curves on Stack A-204 (taken at 143 and 334 hours, respectively) which virtually coincide.

3.2.4.5 Discussion of 200 ASF Results.

Twenty-three stacks were tested at 200 ASF. Of these, 16 met the requirement of an initial voltage of 0.90 v. Six of them were at 30 psia (all 30-psia stacks met the requirement), and 10 at 15 psia. The second requirement of a rate of initial performance degradation not exceeding 0.020 v per 500 hours was met by 13 stacks; 8 failed to meet it, and two may meet it but have not yet been on test long enough. Again, all four eligible 30-psia stacks are included among these 13, but in the 15-psia group only 5 stacks met both requirements. None of these 5 were of flight size.

It has already been pointed out that the evidence gathered does not permit drawing clear conclusions regarding the relative merits of Union Carbide Type-2 anodes and the LAB-40's as a whole, but one may probably differentiate between the various lots of LAB-40's.

First let us pay some attention to the important subject of leakage through the electrodes. Reason would indicate that if the gas pressure on the electrode is higher than the electrolyte pressure on the opposite face of the electrode, leakage of electrolyte through the electrode should be minimal. Both electrolyte and gas pressures are measured, and a method exists for calculating the pressure difference across the electrode at the bottom of the electrode. The convention has been adopted that ΔP is considered positive when the pressure on the gas side is higher. The values for ΔP at various times are shown in Table VI, both for the stacks here discussed (part A of Table VII), and for a few stacks described in the next section (part B of Table VII). These pressure differences should be correlated with the leak rate curves. In general, there is an inverse relation between leak rate and ΔP, but there are numerous instances of disagreement. Leakage through cathodes is, in general, much more severe than through anodes.

TABLE VII

PRESSURE DIFFERENTIAL AT BOTTOM OF ELECTRODE[1]

(Pressure in Equivalent Inches of Water)

Stack No.	Initial Anode	Initial Cathode	100 Hours Anode	100 Hours Cathode	250 Hours Anode	250 Hours Cathode	500 Hours Anode	500 Hours Cathode	750 Hours Anode	750 Hours Cathode	1000 Hours Anode	1000 Hours Cathode	1500 Hours Anode	1500 Hours Cathode	2000 Hours Anode	2000 Hours Cathode	End of Life Anode	End of Life Cathode
A. Data for stacks from Section 3.2.4.2 (preceding)																		
A-015	-15.5	-15.5	-12.7	-12.0	-12.8	-12.3	-13.4	-13.7	-13.6	-13.8	-14.3	-14.7	-14.6	-15.0			6.2	-3.0
A-021	-15.9	-15.7	-12.0	-1.1	-12.1	-1.4	-12.6	-2.5	-12.9	-3.9	-5.1	6.6	8.1	7.1	2.9	7.4	2.0	7.6
A-022	-12.3	-12.6	-15.1	-15.6	-14.4	-15.5	-9.1	12.5	-0.4	10.4	-1.2	10.5					-7.0	-5.1
A-043	-15.4	-15.8	-12.4	-2.9	0.9	-9.0	3.6	-6.6	9.7	-0.3							12.6	-1.5
A-055	-12.5	-12.3	-3.0	4.0	-1.9	-2.0	-0.7	4.0	-0.9	4.7	1.5	8.1	-3.9	-2.1	-2.6	0.0	-4.6	-5.4
A-059	-10.7	-7.8	-6.6	-5.6	-1.4	-4.7	2.3	2.2	-0.8	4.3	5.1	0.7	4.9	0.2	2.2	3.7	-5.8	3.6
A-060	-8.0	-7.0	14.4	-5.7	5.0	-6.6	6.9	-1.8	3.9	-1.7	5.9	0.0	4.4	-1.7			4.4	-1.7
A-066	-11.6	-10.3	-3.7	-6.2	2.7	-5.7	-1.1	-5.9	3.6	0.5	3.7	-0.2	-1.2	-5.3			-1.2	-5.3
A-068	-4.9	-4.8	-3.5	0.1	-6.7	-3.1	-1.0	2.7	-5.6	-2.6	-6.9	-7.3					-7.2	-7.2
A-071	-8.6	-10.3	-6.7	-6.6	-6.9	-6.8	-7.6	-7.2	0.0	-5.3							-6.1	-5.1
A-075	-4.6	1.4	-3.9	2.1	-4.3	1.7	-5.1	0.7	-5.2	0.0							-5.7	0.2
A-077	-10.3	-9.3	-8.7	-10.2	-8.6	-10.7	-7.5	-8.4	0.3	-4.7	0.7	-6.3					-10.0	-12.4
A-081	-12.5	-12.8	-11.9	-13.3	6.9	5.4	7.4	5.9	6.5	5.8							7.2	5.0
A-087	-15.7	-10.4	-9.1	-9.3	4.1	1.9	4.5	-1.8	-1.1	1.9							-4.9	-5.7
B. Data for stacks from Section 3.2.5 (following)																		
A-018	-13.3	-12.8	-13.4	-12.6	-16.8	-15.7	-14.8	-15.0	-14.7	-15.3							-14.2	-14.8
A-035	-14.6	-14.7	-15.2	-15.4	-15.8	-16.6	-16.1	-17.2	4.5	0.0							4.6	-0.1
A-061	-11.0	-11.5	-17.8	-3.1	2.3	10.2	12.3	2.6	13.1	6.3	13.1	6.4					13.7	8.0
A-053	-10.7	-7.4	-5.0	-1.5	-5.0	-1.4	-2.1	-1.0									-0.7	-0.6
A-072	-9.4	-9.1	-9.9	-9.5	-9.6	-8.8											-9.3	-8.5
A-051	-12.5	-12.6	1.0	-1.0	7.4	2.3	11.3	1.8	3.7	-6.3	0.6	-7.2	2.5	2.3			2.5	2.5

[1] Positive value indicates higher pressure on gas side of electrode.

First, consider anodes: The T-2's are intermediate between certain groups of LAB-40's. They leak less than the early lot of LAB-40's, but more than the A-II and B-II-4 versions. As to cathodes, the old LAB-40's are poor, the A-II's are next, with the B-II-4's comparable. The 143's leak the least. Notable exceptions are the A-061 cathode, which showed high leakage even with a positive ΔP, and the A-072 cathode, which showed no leakage with a large $-\Delta P$. Unfortunately, there appears to be an inverse relation between desirable electrical performance and leak rate. The poorest stacks—from an electrochemical viewpoint—leak least, and the best stacks weep copiously. All of the stacks with lives >1500 hours had fairly high leak rates for the cathodes, while the poorest stacks, A-081 and A-087, showed only low leakage through the cathode.

As mentioned in the discussion of Stack A-015, the internal resistance of most of the stacks is very low and does not vary with age. The stack voltage varies somewhat with temperature and electrolyte normality, but at least in 14 N KOH the temperature dependence is much stronger than the concentration dependence. It is therefore very important to keep the temperature constant.

Consider now the lifetimes of LAB-40 electrodes: Among these the LAB-40-A-II appear best, either as all LAB-40 stacks or in hybrids (Stacks A-055, A-059, and A-060). Next probably are the older, undesignated LAB-40's, then come the B-II-4's (Stacks A-077 and A-079) which might be better than the others mentioned, but there is not yet enough evidence. The LAB-40 (-143, A and B types) are clearly the worst.

3.2.5 Tests under other Conditions.

3.2.5.1 Tests at 100 ASF.

Three tests at this load level were inconclusive except to show that a ChemCell cathode was not acceptable. The two other tests were terminated for reasons affecting, but not due to, stack performance.

Stacks A-018 and A-035 differed from the usual stack construction in that they both were furnished with additional current collectors. A gold-plated 5Ag7-4/0* screen was pressed against the face of each electrode and was held under the customary silver frame in silver epoxy.

* Exmet Corporation, Bridgeport, Connecticut designation.

Stack A-018 (Figs. 19, 52, and 86) has a questionable operating history. The unit suffered several accidental shutdowns which may have had a deleterious effect. The stack initially developed 0.940 v at 100 ASF, and remained at that level for some 520 hours. At that point it suffered its second shutdown because of low electrolyte level, and thereafter started to decline. It had dropped to 0.910 v at 700 hours, and at 708 hours it again shut down due to low electrolyte level. Subsequently the potential dropped to 0.75 v at 804 hours. At that time the load was reduced to 50 ASF, and the potential rose to 0.880 v and held steady there. But at 828 hours, the test was concluded to permit extensive modification of the test position preparatory to receiving the next stack.

Autopsy of A-018 revealed that on both electrodes the Teflon could be peeled off readily with tweezers. The back of the cathode was partially wet, the back of the anode was very wet, and the anode was probably drowned. The apparent separation of the layers comprising the LAB-40 electrodes caused some concern (this was the first stack of this type which had been shut down and autopsied). Subsequent process modification by the vendor led to improved bonding of the backing to the active material.

Stack A-035 (Figs. 22, 55, 90, and 91) used a ChemCell electrode. The test was run at 100 ASF because measurements made during the previous reporting period made us doubt whether this electrode could long carry the desired 200 ASF. Even at 100 ASF the performance was not good. This stack held its starting potential of 0.910 v for 200 hours and then dropped gradually to 0.820 v at 598 hours, when it was removed for washing and drying. It showed little improvement when it was returned to test (0.83 v), and the polarization curves just before and after washing and drying (Fig. 91) indicate a continuing decline. Performance continued to drop off gradually to 0.81 v at 780 hours, then more steeply. The stack was removed from test at 0.730 v after 830 hours.

The stack had suffered only one accidental shutdown, caused by the general power failure on February 16, 1967, at 603 hours. At times the cathode performance seemed sensitive to the O_2 pressure. The leak rate through the ChemCell cathode (Fig. 55) was substantial. The stack showed a KOH flow of 170 ml/min. at 1.5" KOH head, and its electrical resistivity was about 0.5 milliohm·ft^2, rising slightly with age. Polarization curves (Figs. 15 and 16) show that the stack never fell within the envelope of "normal" polarization curves. The initial curve, taken when the stack had been on test only 3 hours, was poor even then.

Stack A-061 (Figs. 29, 62, and 98) was an excellent stack. It maintained its initial potential of 0.97 v for some 270 hours, held 0.96 v to 820 hours, and was steady at 0.95 v when (at 1,174 hours) the test had to be concluded. The unit had overheated (as was apparent from the melting of the Lamport polypropylene separator), and failed to support reasonable load. The anode side of the unit, showing the fused central portion of the separator, is displayed in Fig. 14. Autopsy revealed that here was another case of failure due to the disintegration of the nylon KOH separator. Only traces of the separator were found in the cell. The middle three inches of the electrodes were in intimate contact. The carbon from the Union Carbide T-2 anode stuck to the cathode; otherwise, the anode showed no signs of damage, even though the polypropylene separator in back of the anode had melted in the central portion. The LAB-40 cathode also did not show any effect directly attributable to the overheating, except that the Teflon backing had a quilted appearance caused by the impression of the separator in the oxygen space in the heat-affected zone. There were also a few isolated small regions along the sides of the cathode, within an inch of the edge, where the Teflon appeared to have bulged away from the cathode in the areas where there are openings in the separator.
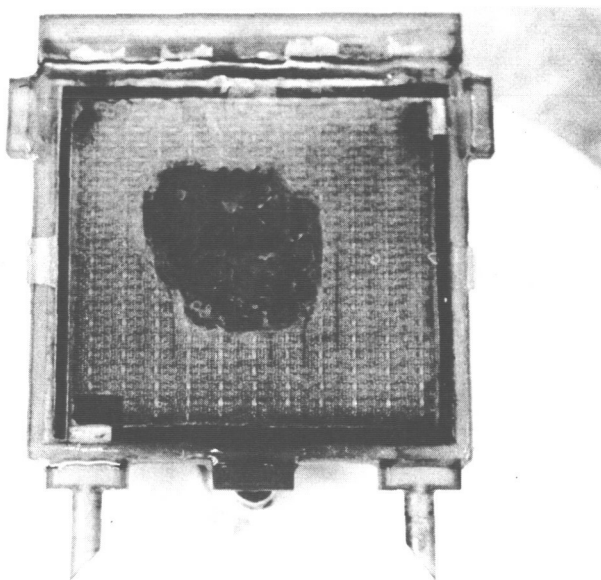
D-3463

Fig. 14 - Anode Side of Stack A-061 after Removal from Test.

The polarization curves for Stacks A-018 and A-061 showed good performance initially. Unfortunately, the first test was vitiated by test stand mishaps, and the second one by overheating due to a materials problem. But the rate of voltage decay had been less than 0.020 v/1000 hours, and a polarization curve near the end of life showed only a minimal drop-off in performance.

3.2.5.2 Tests at 300 ASF.

Stack A-053 (Figs. 25, 58, and 94) held its initial potential of 0.86 v for about 120 hours. Thereafter, the voltage dropped fairly uniformly and steeply to reach 0.78 v after 406 hours. At 454 hours and 0.76 v, the load was reduced to 200 ASF; the potential rose to 0.84 v but it dropped again rapidly to reach 0.76 v at 600 hours, and was shortly thereafter removed from test. Leakage was only moderate, but the polarization curve reveals a great drop in capability. Since this poor performance might have been due to some reason other than inherent electrode properties, a similar test was run with Stack A-072 (Figs. 34, 67, and 103). This stack behaved even worse. It dropped from an initial 0.87 v to 0.745 v at 236 hours. Reducing the current to 200 ASF brought the potential back to 0.86 v; two days later, however, at 284 hours the stack was shut down with a potential of 0.67 v.

Stack performance at 300 ASF was clearly poor; the electrodes degraded rapidly. Fig. 15 shows the envelope of some 13 stacks run at 200 ASF (from Section 3.2.4.1) with the stacks from this Section (3.2.5.2) and Stack A-035 added. The "initial" readings for both Stacks A-053 and A-072 were taken at about 100 hours of life, at which time they had already fallen below the comparable norm. Five hundred hours later (Fig. 16), A-072 was dead, and A-053 had been running at 200 ASF for 150 hours and was just about dead. Stack A-053 contained all LAB-40 electrodes, while A-072 was a hybrid.

The samples in the preceding two sections are too limited to permit any conclusions to be drawn about electrode quality, except in the case of the ChemCell cathode.

3.2.5.3 Tests under a Similated System Load Cycle

Four hybrid stacks were tested under the load cycle described and rationalized at the end of Section 3.2.2. Three of the tests are still in progress; the results to date are summarized in Table VIII. But, as is obvious, these figures show only part of the story; most encouraging are the long lives and low degradation rates observed in this system simulation.
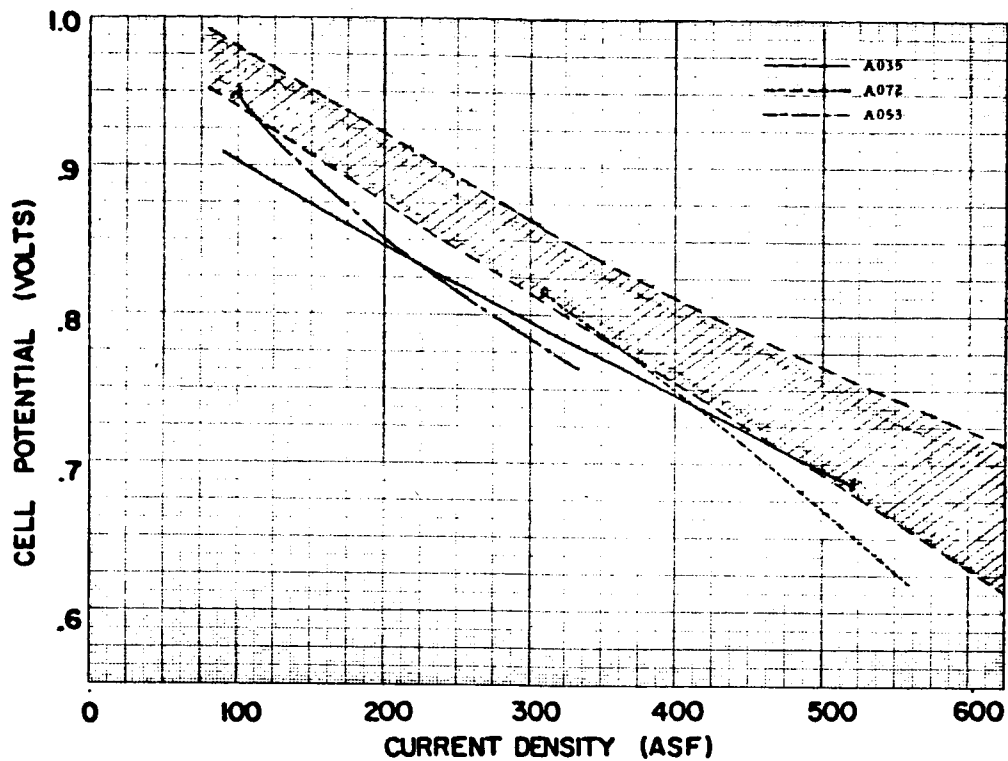
D-3468

Fig. 15 - Envelopes of Initial or Early Polarization Curves Showing Subaverage Performance of Cells A-035, A-053, and A-072.
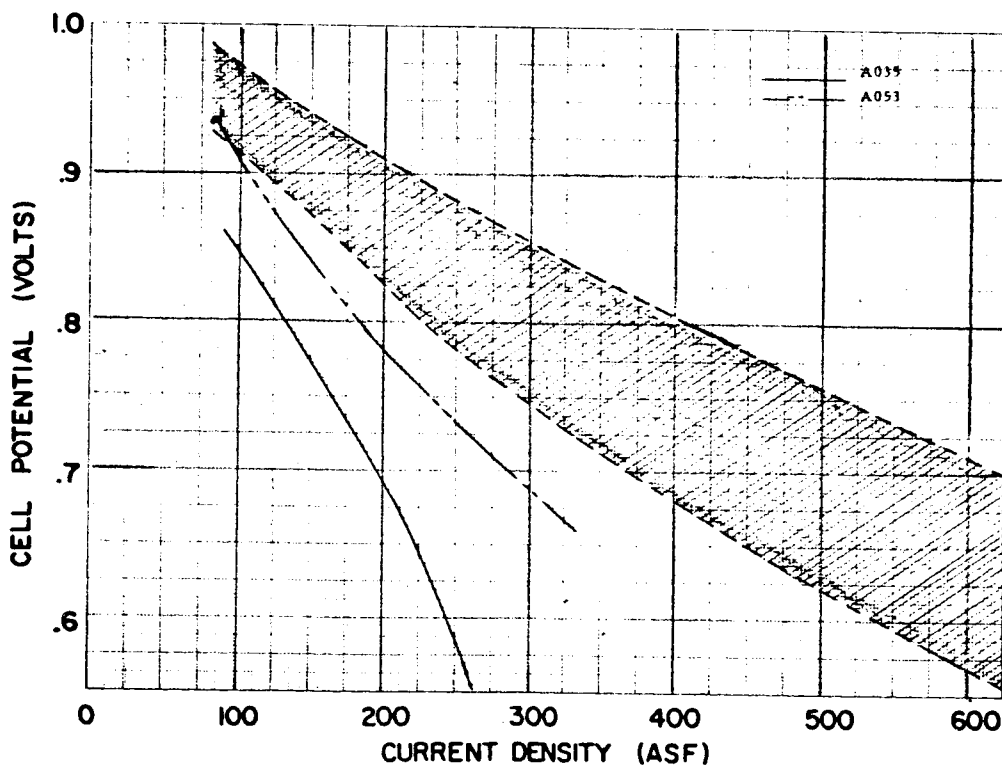
D-3469

Fig. 16 - Envelope of Individual Polarization Curves; 500 Hours Later than Fig. 15.

-46-

TABLE VIII

PERFORMANCE SUMMARY,
SMALL STACKS UNDER SIMULATED SYSTEM LOAD

| Stack No. | Current Density (ASF) | Cell Voltage at | | | | | | | | | As of 0900, 1 July 1967 | | Life-time Hrs |
		Peak	100 Hrs	300 Hrs	500 Hrs	700 Hrs	900 Hrs	1000 Hrs	1500 Hrs	End of Life	V	Hrs	
A-069	124	.920	.920	.920	--	--	--	--	--	.920	--	--	356*
	200	.880	.880	.870	--	--	--	--	--	--	--	--	
A-073	124	.925	.925	.925	.920	.925	.920	.910	.920		.910	1710	On test
	200	.880	.880	.880	.880	.880	.870	.860	.860				
A-084	124	.940	.925	.920	.920	.915	.915	.915			.910	1191	On test
	200	.880	.875	.860	.865	.860	.855	.850					
A-090	124	.930	.930	.925	.920	.920	.915	.910			.905	1394	On test
	200	.885	.885	.880	.875	.875	.865	.860					

* Shutdown because of internal mechanical damage following pump failure.

Stack A-069 (Figs. 32, 65, and 109) was running well and maintaining its initial potential when the KOH pump failed at 334 hours (during the night). The electrolyte looked quite black and must have overheated, because in portions of the system it had solidified. After the pump was replaced it proved impossible to circulate any KOH through the system, so all lines were flushed with distilled water under pressure, and finally the cell was reactivated. But the next morning it was on open circuit again, and the system shut down. Much electrolyte had drained into the anode collecting bottle, and there was a substantial leak through the anode. Furthermore even on open circuit only a 0.60 v potential was maintained. The test was therefore concluded at 356 hours.

The other three units behaved normally, except that Stack A-084 was received with a frame leak (possibly a tear) along the top edge of the anode. To repair this, a slit was cut into polysulfone end plate, epoxy was poured in, and the end plate resealed. That seemed to be an adequate repair, as shown by the performance of the stack. But now the actual active electrode area was, of course, somewhat smaller than standard—0.106 instead of 0.12 ft^2.

There is apparently at least one difference between the cyclically operated stacks and the others tested. The anodes in the former seem to leak more. One cannot tell without further testing whether this is due to the mode of operation or to the reduced electrolyte concentration. Unfortunately, we were unable to obtain the data needed to calculate the pressure differentials across the electrodes on the older test stands used in this experiment. But in any case, the higher leak rate does not seem to affect the electrical performance of the stacks adversely.

3.2.5.4 Thermal Cycling.

The disturbing observation had been made on Stack A-018 and certain other early units that the Teflon backing tended to separate from the active portion of the LAB-40 electrodes. Because of the various accidental shut downs which beset us in the early stages of this program, it could not be established from the information available whether this separation was due to age or due to cycling. The subject was also discussed with representatives of American Cyanamid Co., who assured us that the later lots of electrodes had been improved in that respect. Hence, a test involving deliberate thermal cycling was undertaken.

Stack A-051 (Figs. 24, 57, and 93) was run for about 16 hours (overnight) at 200 ASF. Each morning it was placed on open circuit, the heater shut off, and water circulated through a Ni cooling coil immersed in the electrolyte

tank, so that the unit was brought to between room temperature and 35°C by about noon. Then the cooling water was turned off, the heater turned on again, and the system brought back to 90°C by late afternoon; at which time, the load was reapplied for the next load cycle.

Initially, the stack showed 0.895 v, but from 60 to 133 hours on load (163 hours on test), the potential was 0.90 v and then it began to drop slowly. After 32 thermal cycles (1,123 hours on test, 944 hours at 200 ASF), the stack still showed 0.825 v. At that time cycling was discontinued and the stack performance was followed at 200 ASF continuous duty. The potential continued to drop at about the same rate as it had done while on the cycling test, falling below 0.78 v after some 1,370 hours under load; 1,527 hours on test. At 1,452 hours (0.73 v) dilute gas tests showed the anode performance had dropped off much more than the cathode, but rejuvenated the stack to give 0.88 v. But this improvement did not last, and at 1,478 hours (0.80 v), the stack was removed for washing and drying. Upon return to test, it showed only 0.69 v, so the load was reduced to 100 ASF, giving 0.855 v. By 1,498 hours, the potential was down to 0.78 v, and after 1,522 hours under load (1,678 hours on test), the unit was removed at 0.70 v. Just before removal, another set of dilute gas tests showed the anode to be almost dead; when the dilute H_2 was substituted for the 100% H_2, the stack potential dropped to zero in about 70 seconds.

While the stack was still functioning well (at 805 hours under load), it was used to obtain interrupter readings at different temperatures and thus allow estimating the temperature dependence of both the voltage and the electrical resistivity. The curves are shown in Fig. 17. These are the values obtained for the resistivity, as follows:

Temperature (°C):	36	45	65	90
Resistivity (milliohms·ft^2):	0.84	0.60	0.47	0.34

The temperature dependence of the resistivity is not linear, nor even simple, and was therefore not evaluated analytically. The temperature dependence of the potential is also not simple, but it is most marked at the lower temperatures; less so at the higher end of the scale of interest. To a first approximation, one may take $\Delta V/\Delta T = 3$ mv/°C between about 50° and 90°C.

A series of experiments has been scheduled to determine more precisely the dependence of voltage on temperature and pressure at various current densities.
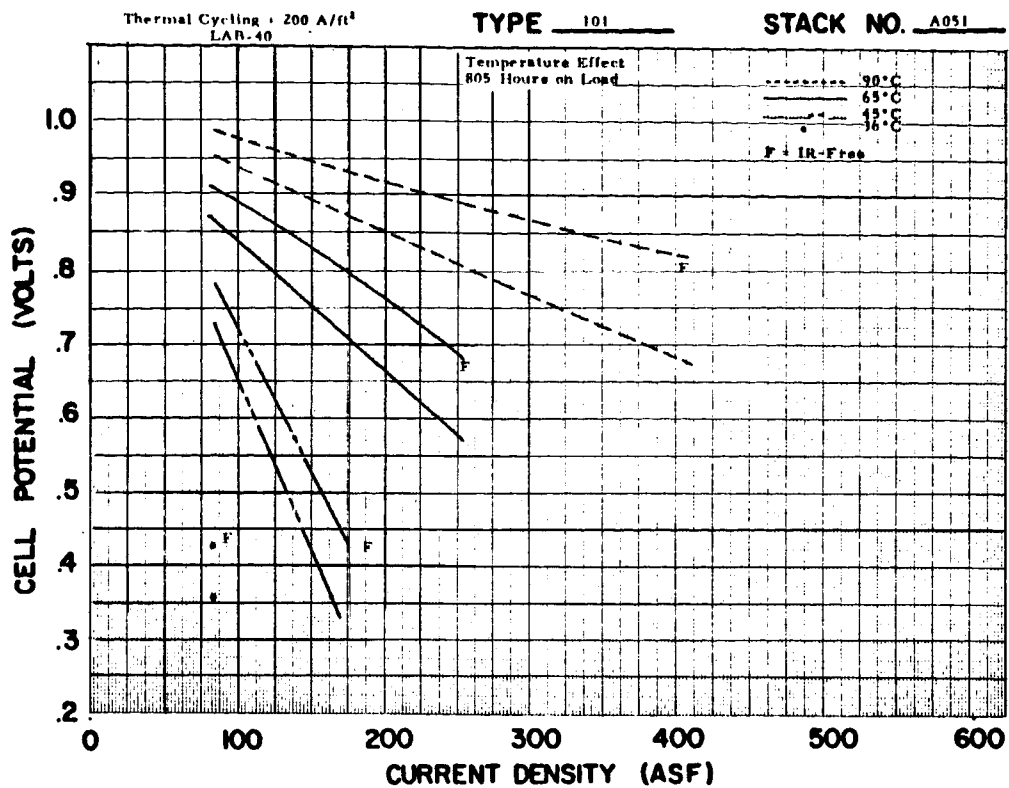
Fig. 17 - Polarization Curves of Stack A-051; 840 Hours under Load, as a Function of Temperature.

3.2.6 Time-Voltage Curves, Time-Leak Rate Curves, and Individual Polarization Curves for All Stacks Described.
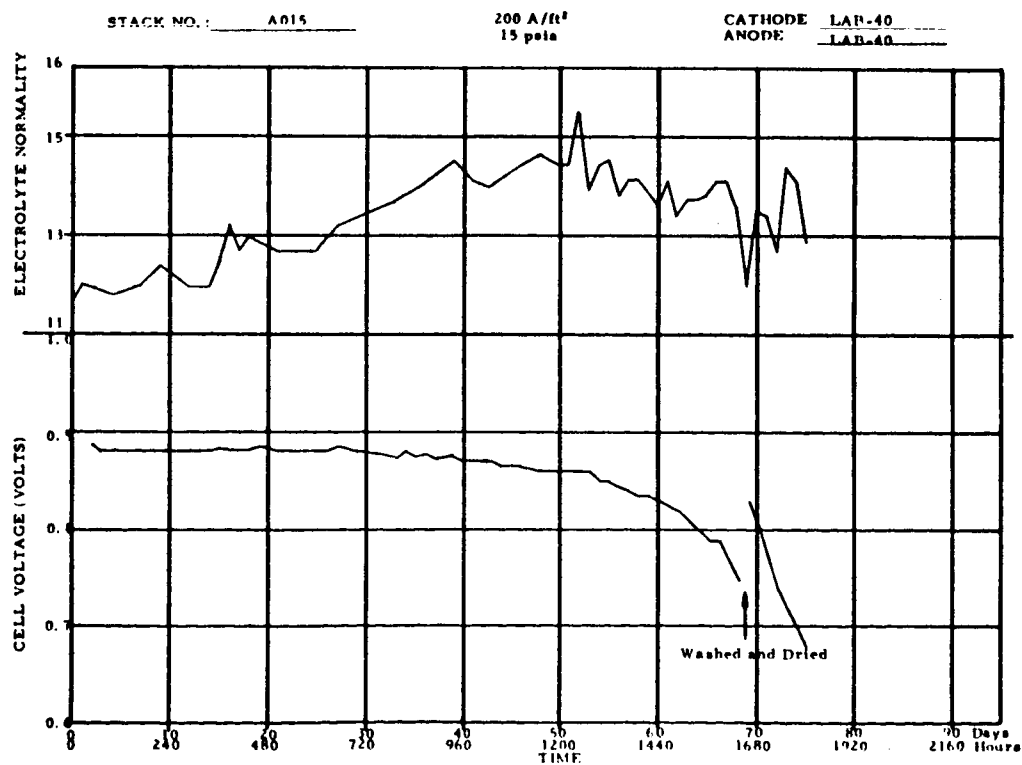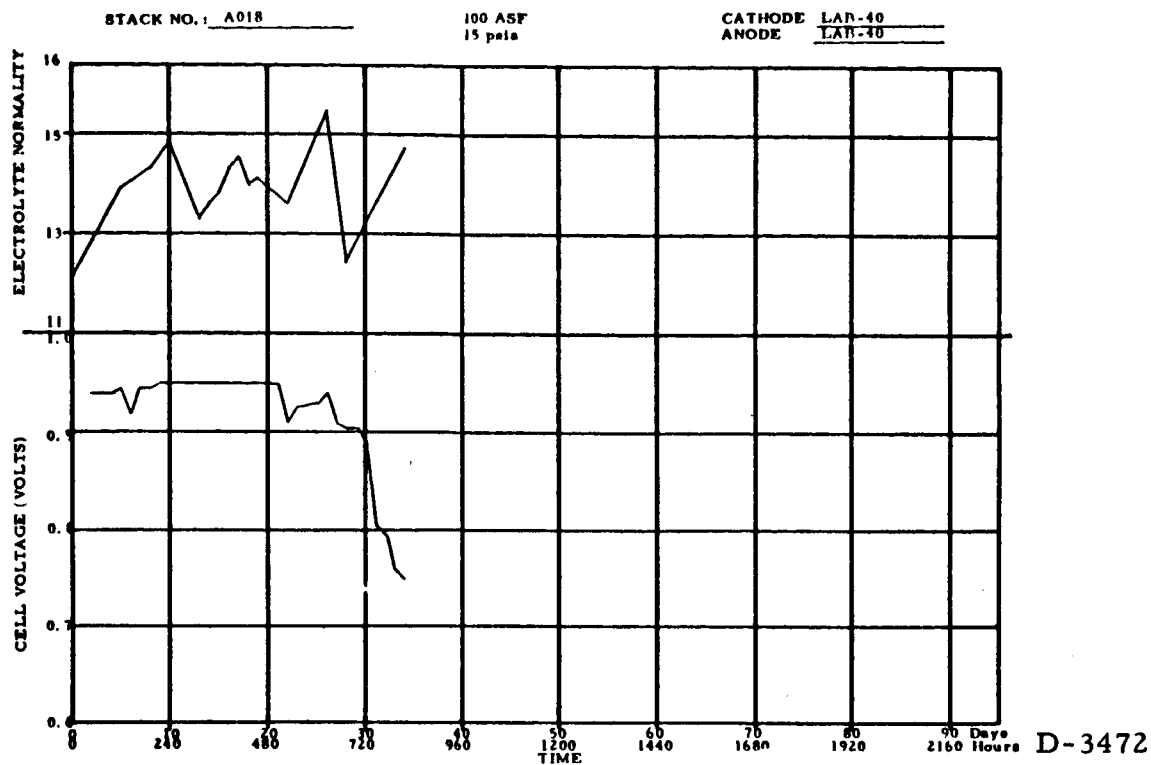

Fig. 18 - Time Versus Voltage - Stack A-015.

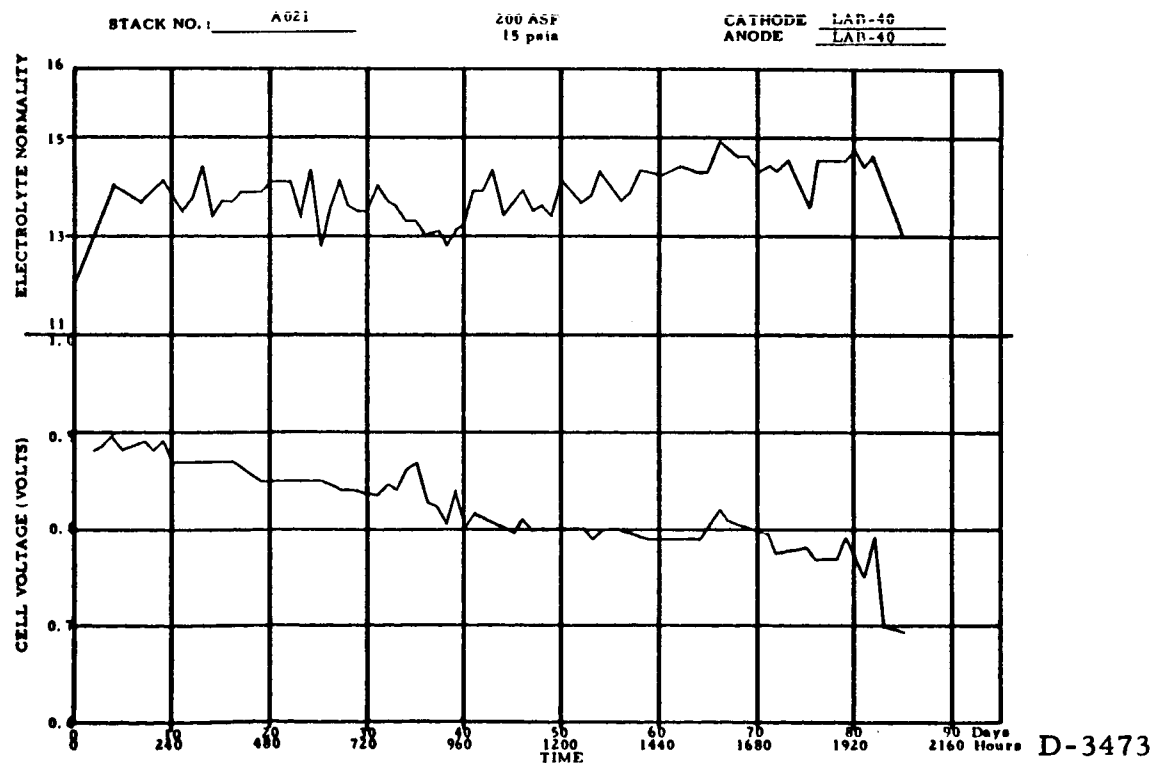D-3472

Fig. 19 - Time Versus Voltage - Stack A-018.

D-3473
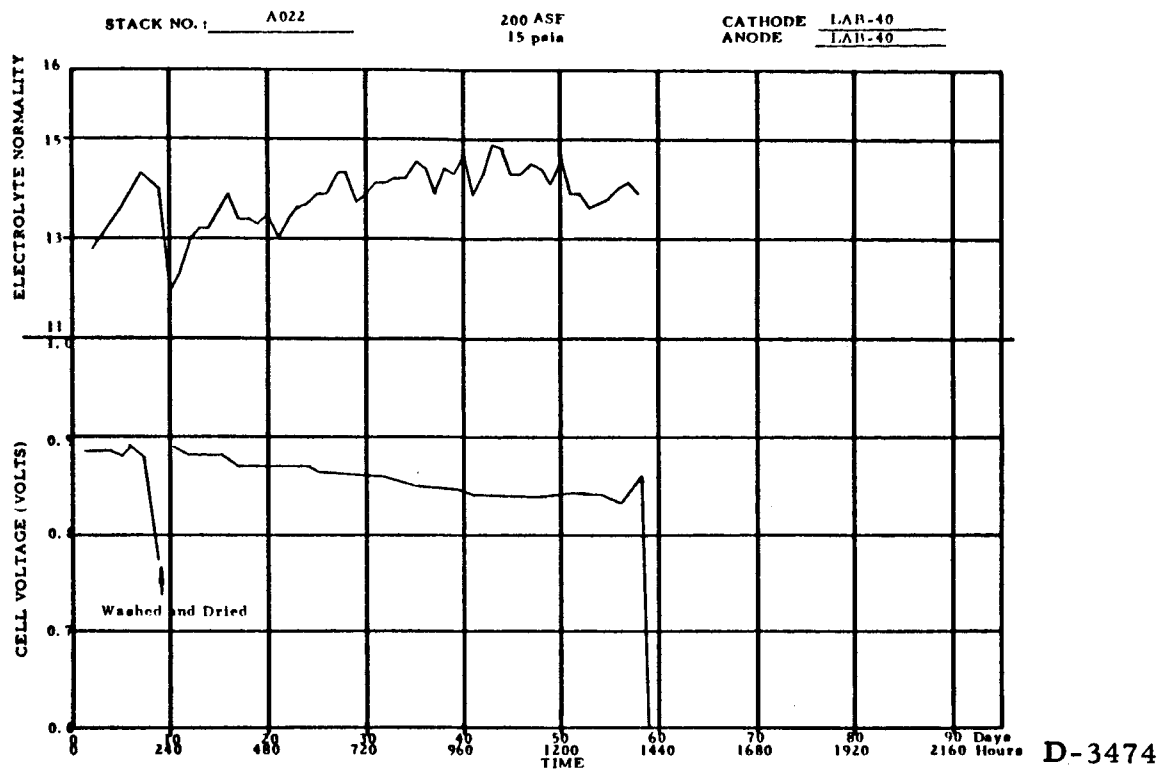
Fig. 20 - Time Versus Voltage - Stack A-021.

-51-

D-3474
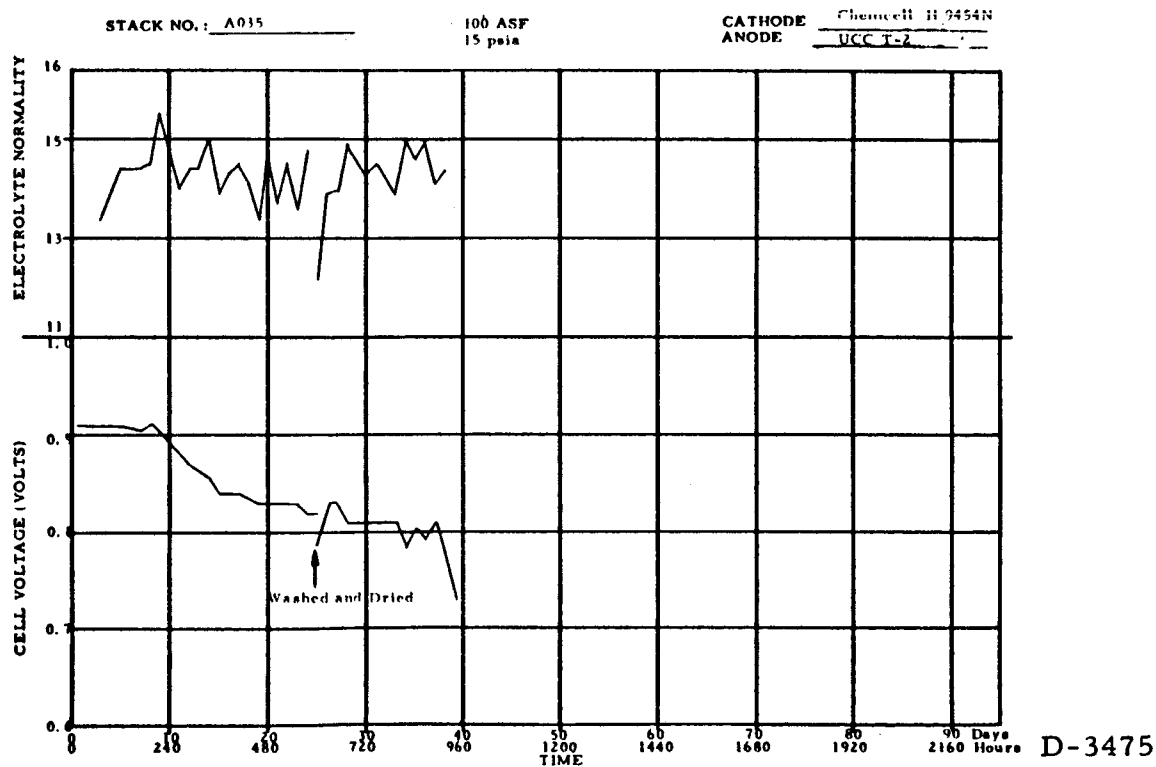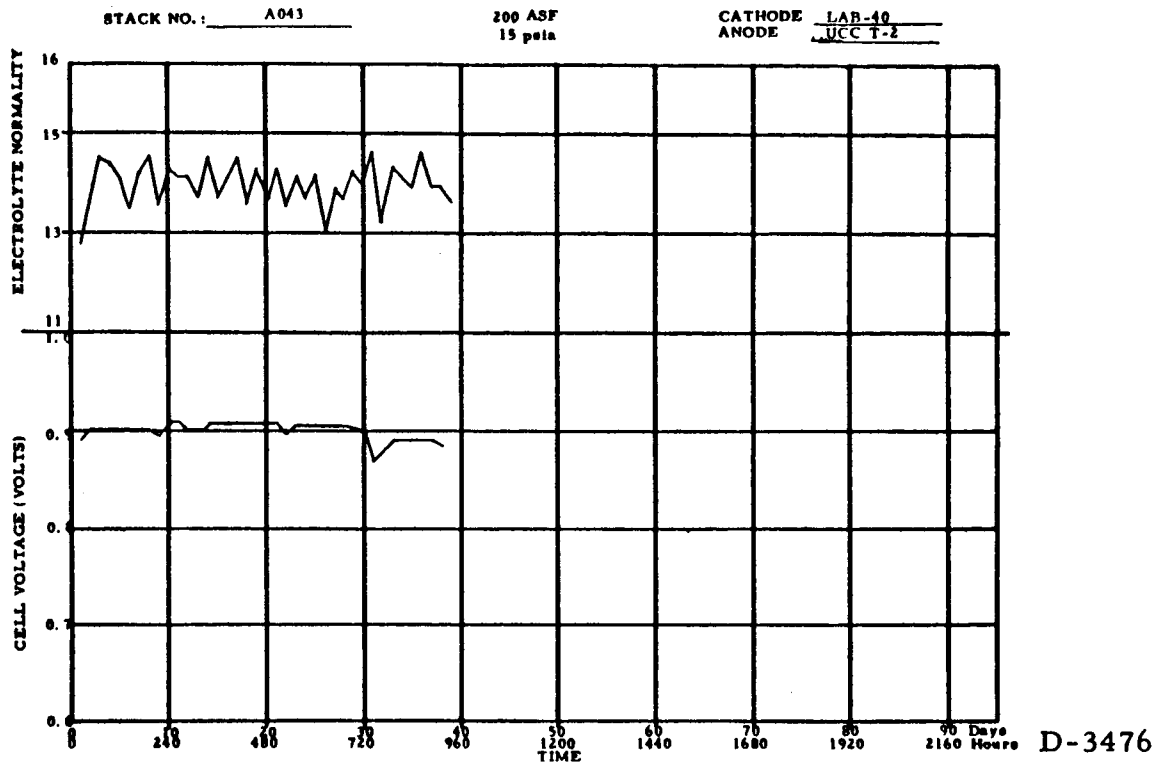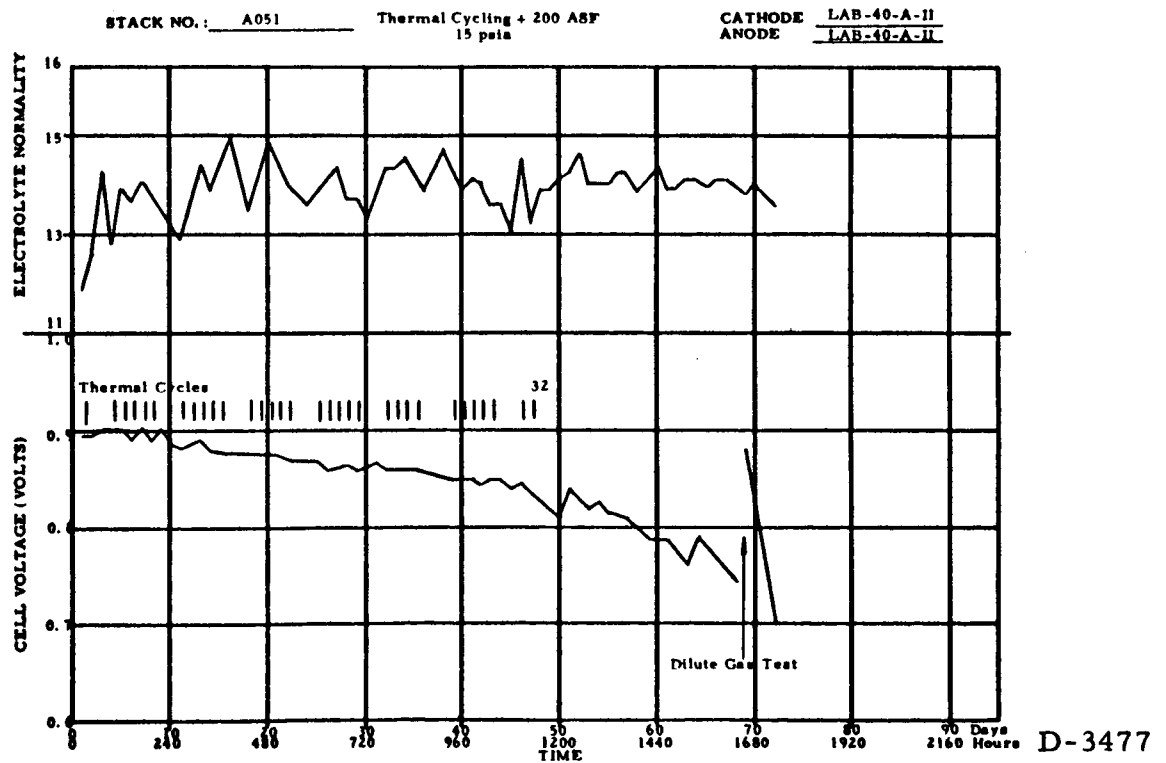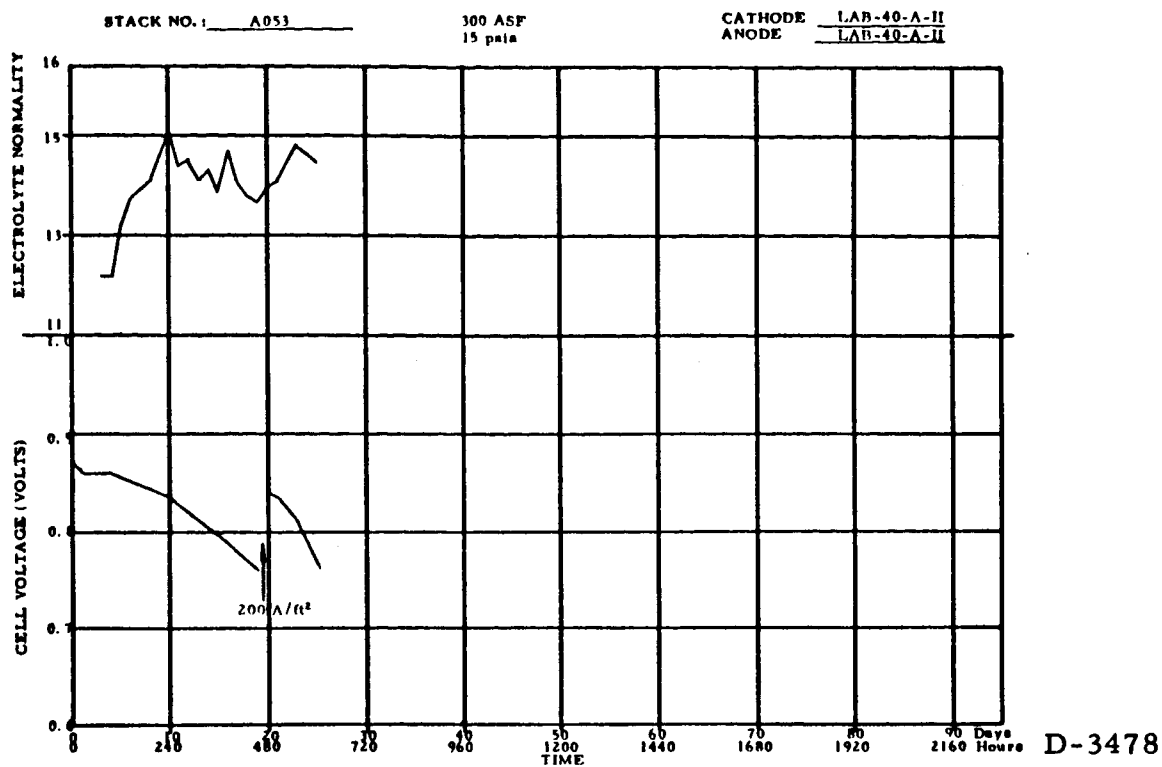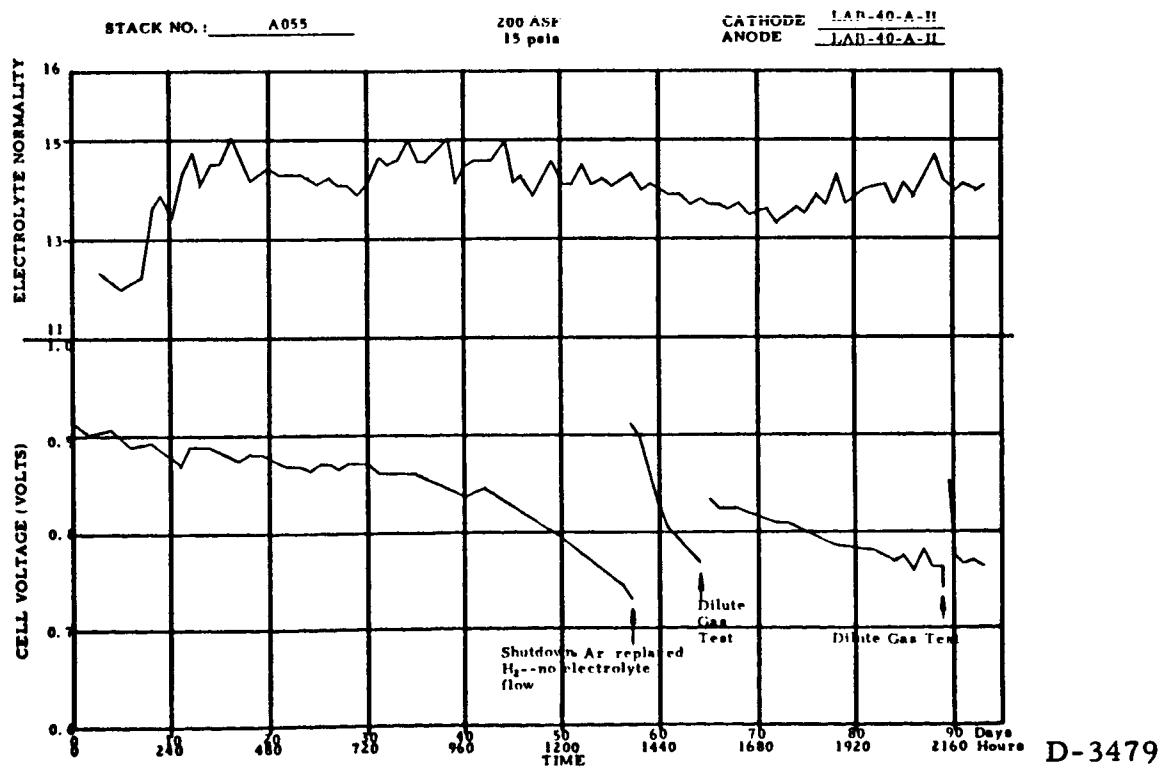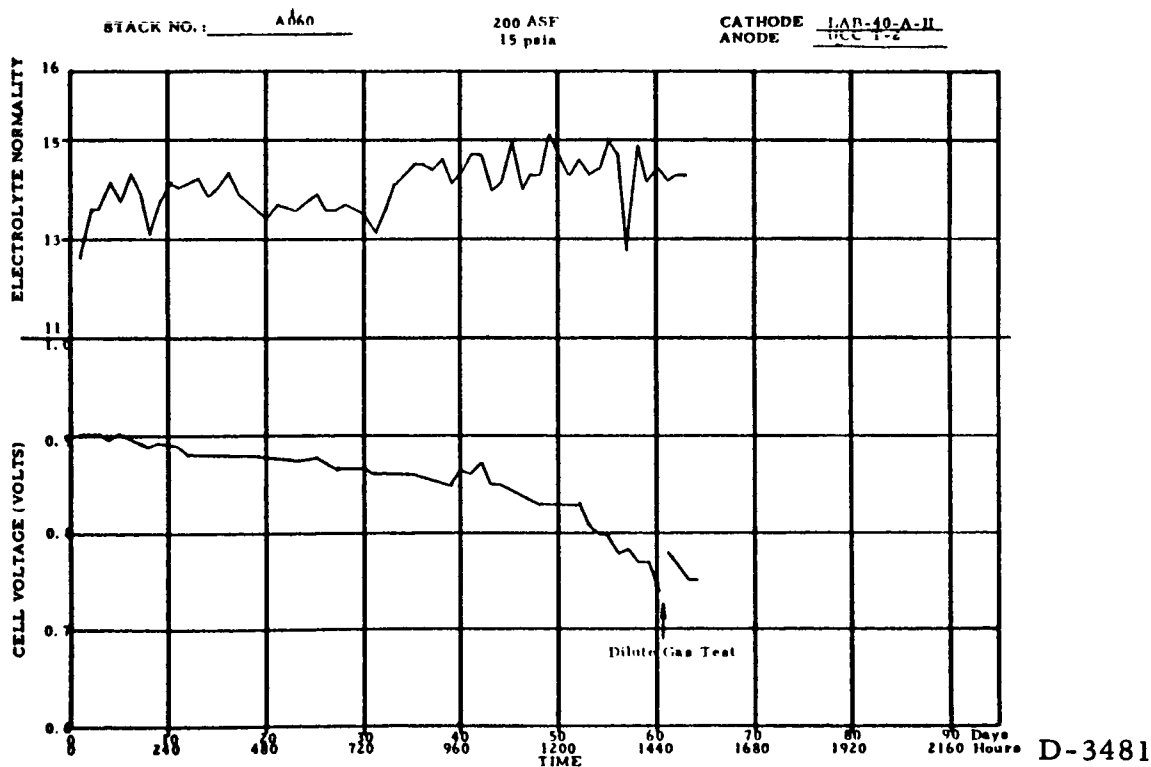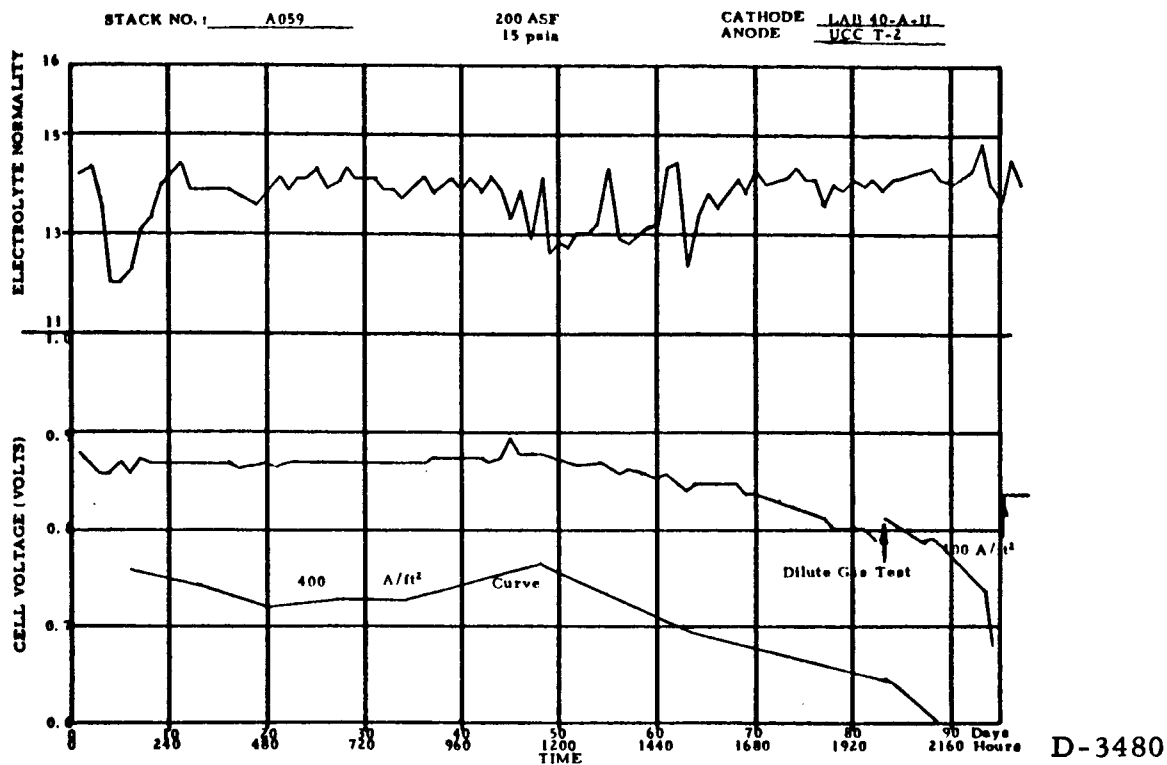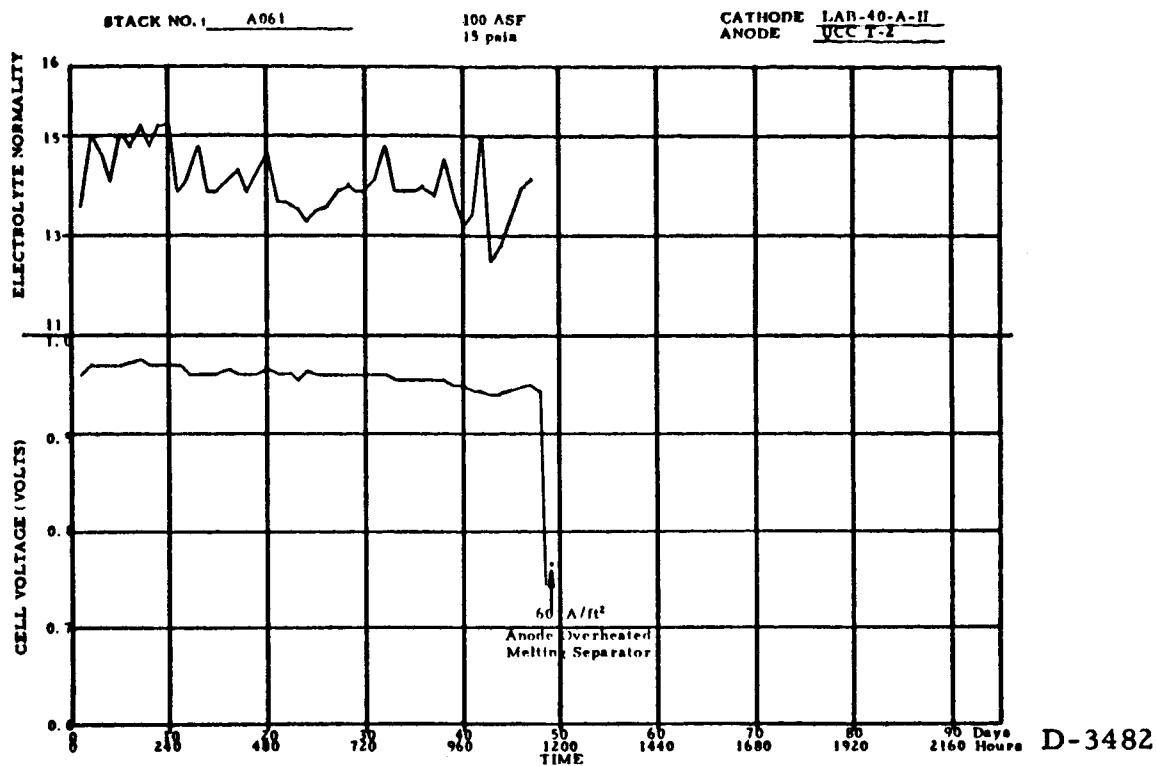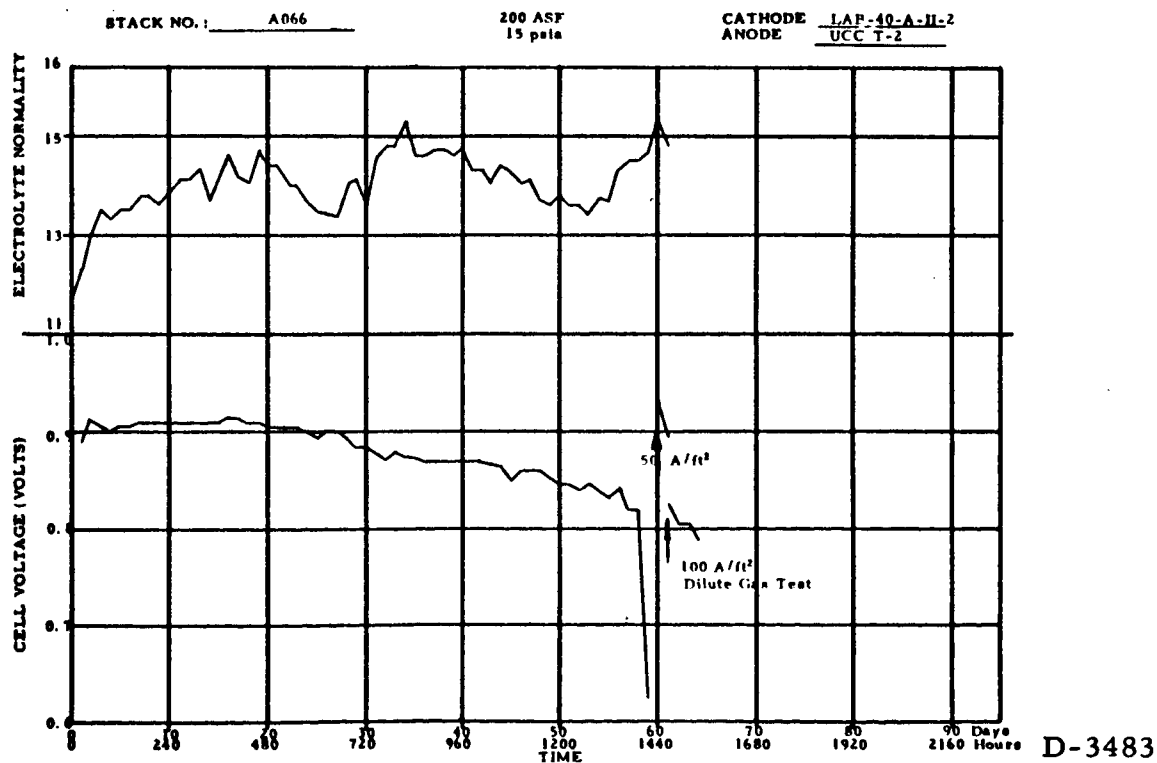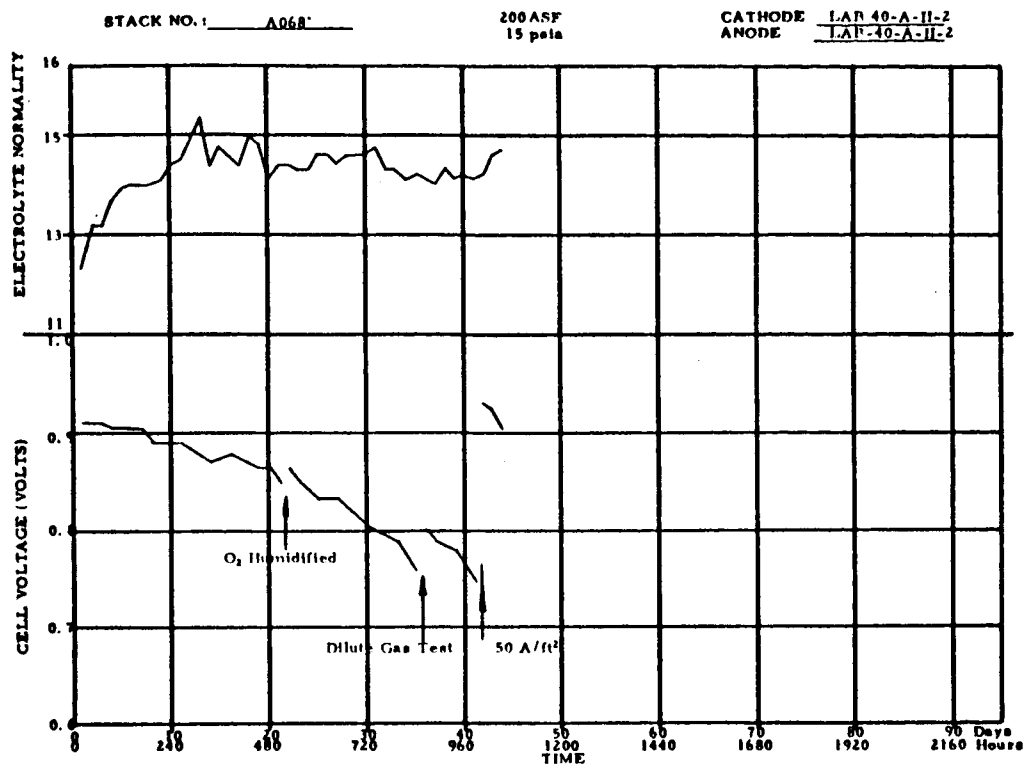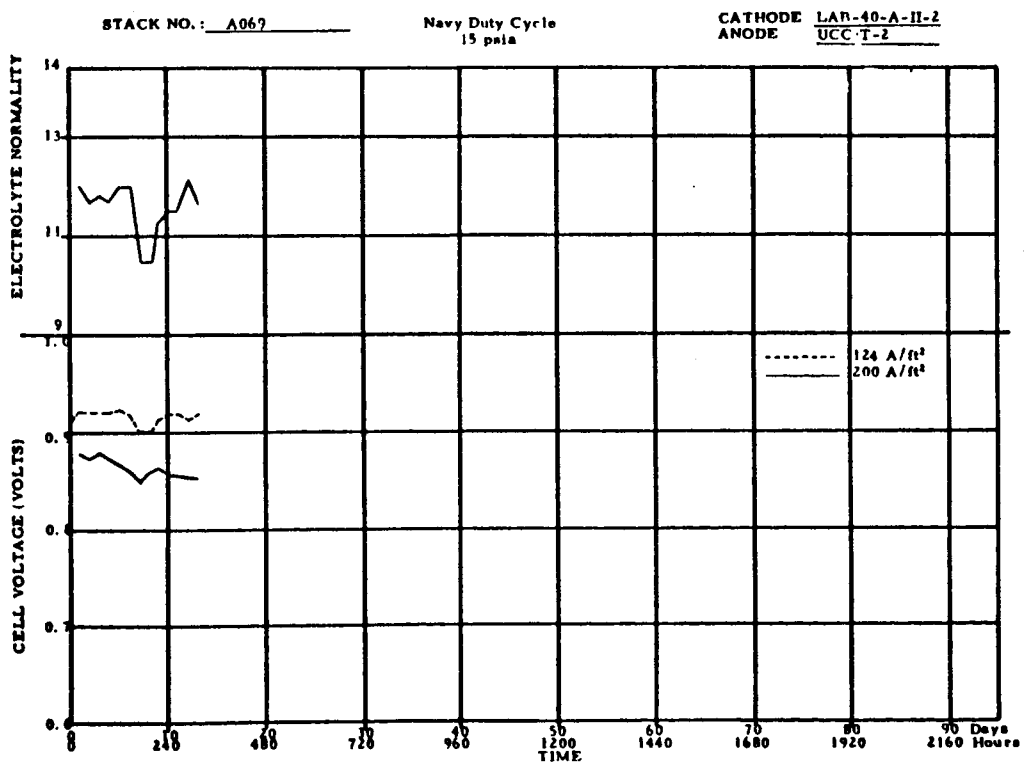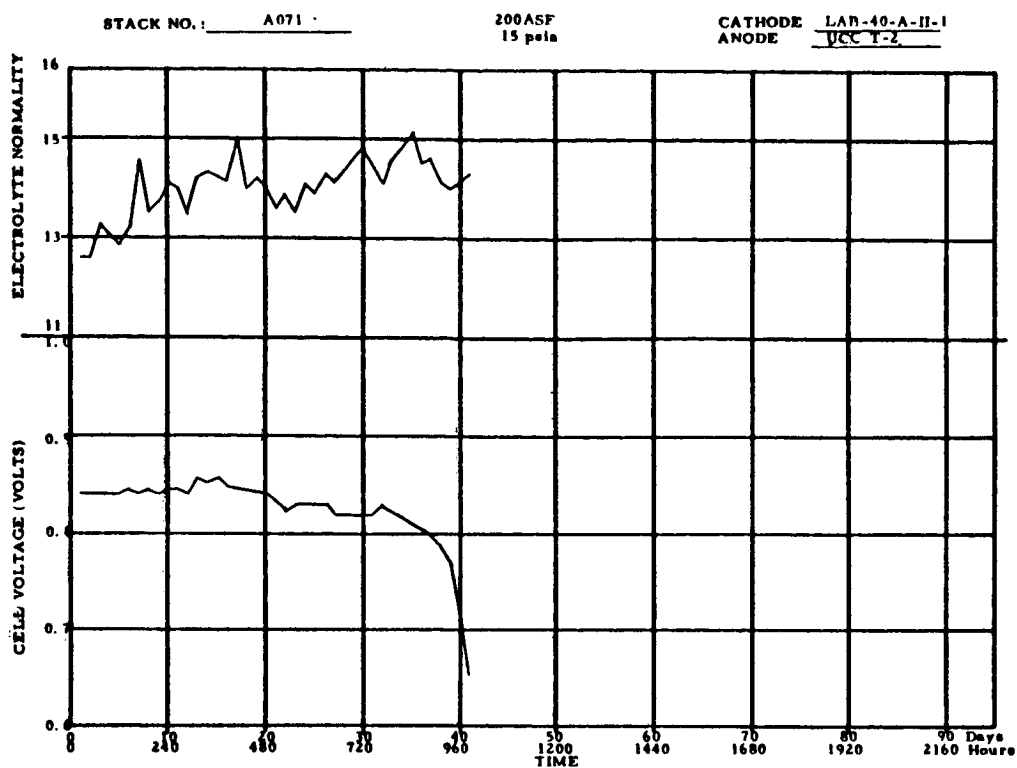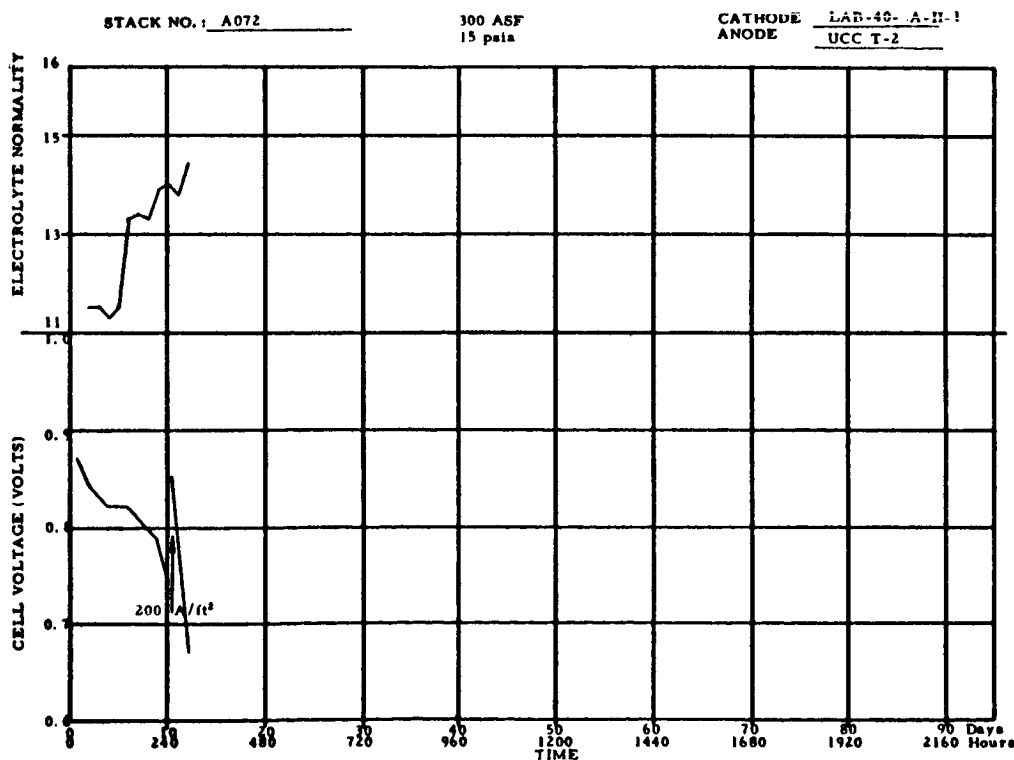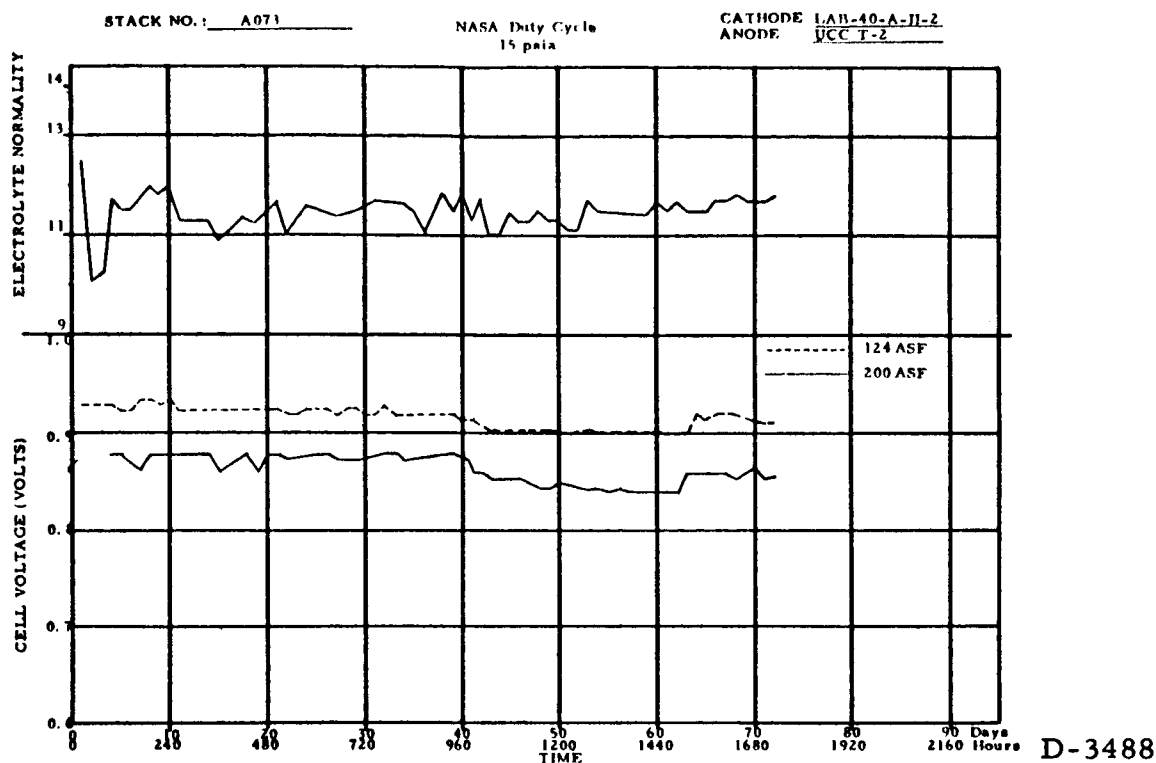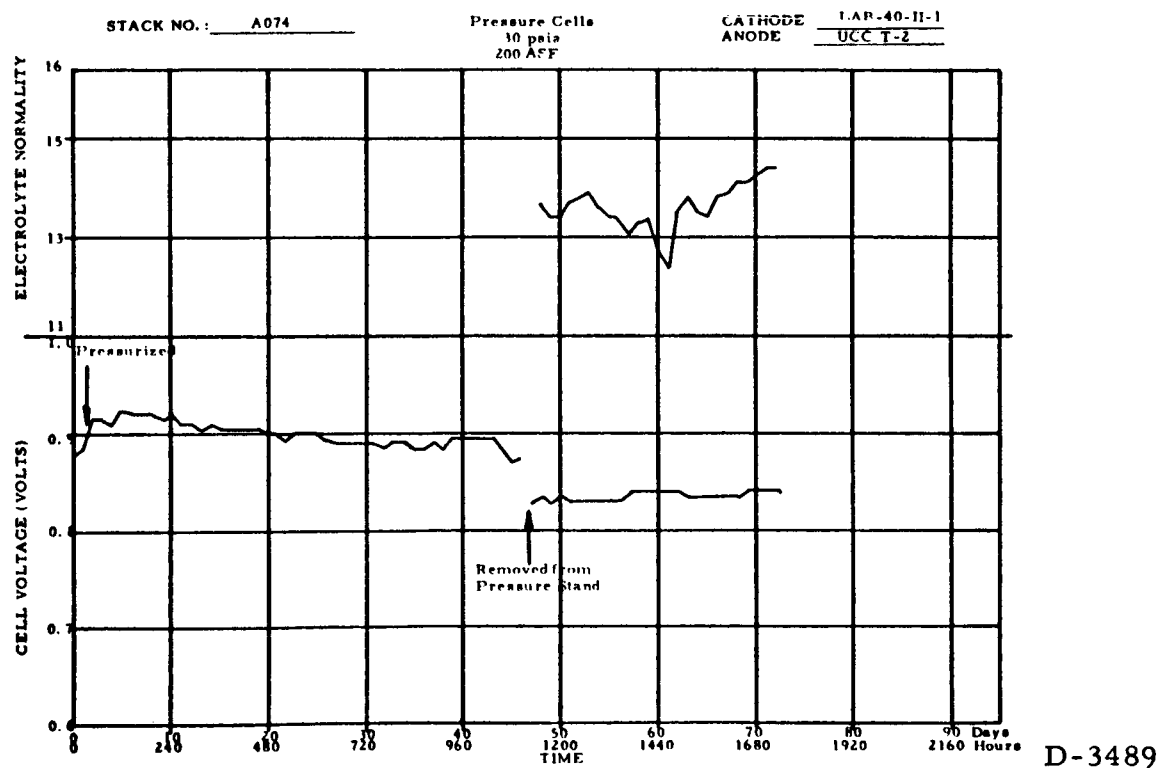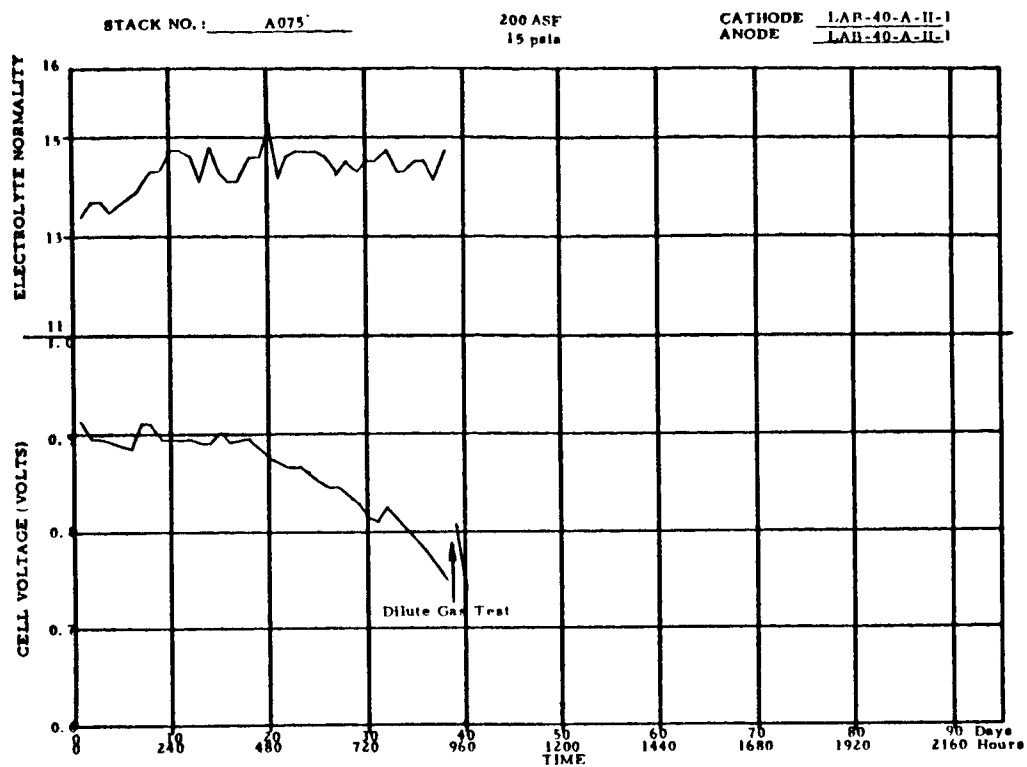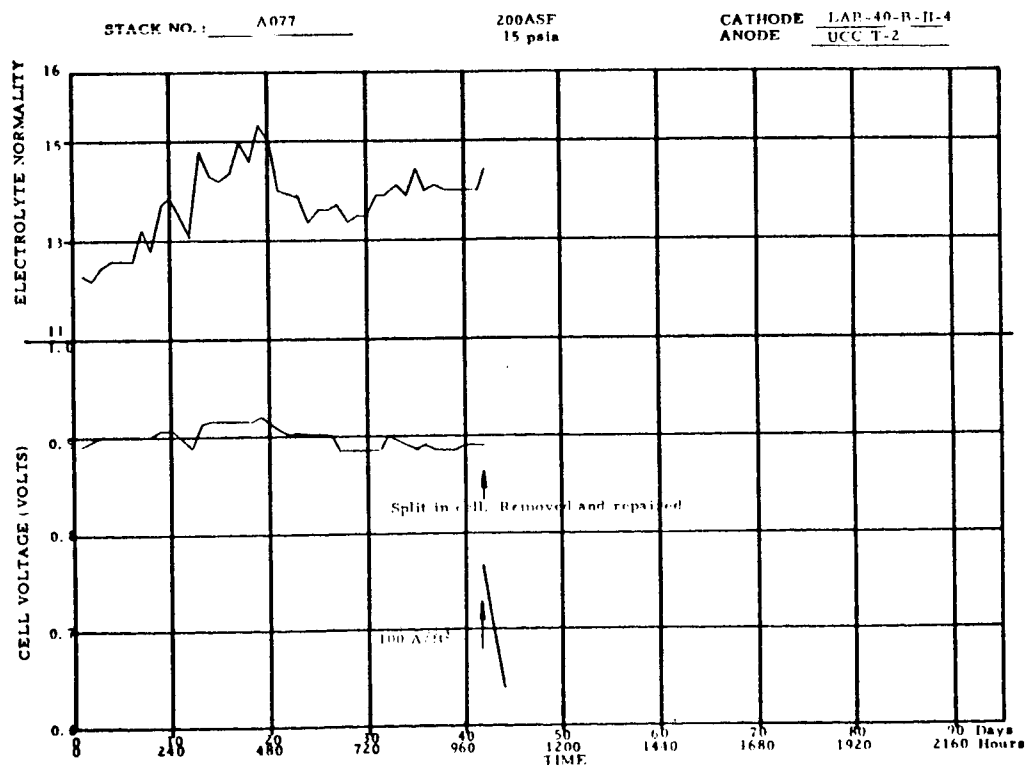
Fig. 21 - Time Versus Voltage - Stack A-022.

D-3475

Fig. 22 - Time Versus Voltage - Stack A-035.

-52-

ELECTROLYTE NORMALITY

CELL VOLTAGE (VOLTS)

TIME

D-3476

Fig. 23 - Time Versus Voltage - Stack A-043.

ELECTROLYTE NORMALITY

Thermal Cycles   32

CELL VOLTAGE (VOLTS)

Dilute Gas Test

TIME

D-3477

Fig. 24 - Time Versus Voltage - Stack A-051.

STACK NO. : A053   300 ASF   CATHODE LAB-40-A-II   ANODE LAB-40-A-II

D-3478

Fig. 25 - Time Versus Voltage - Stack A-053.

STACK NO. : A055   200 ASF   CATHODE LAB-40-A-II   ANODE LAB-40-A-II

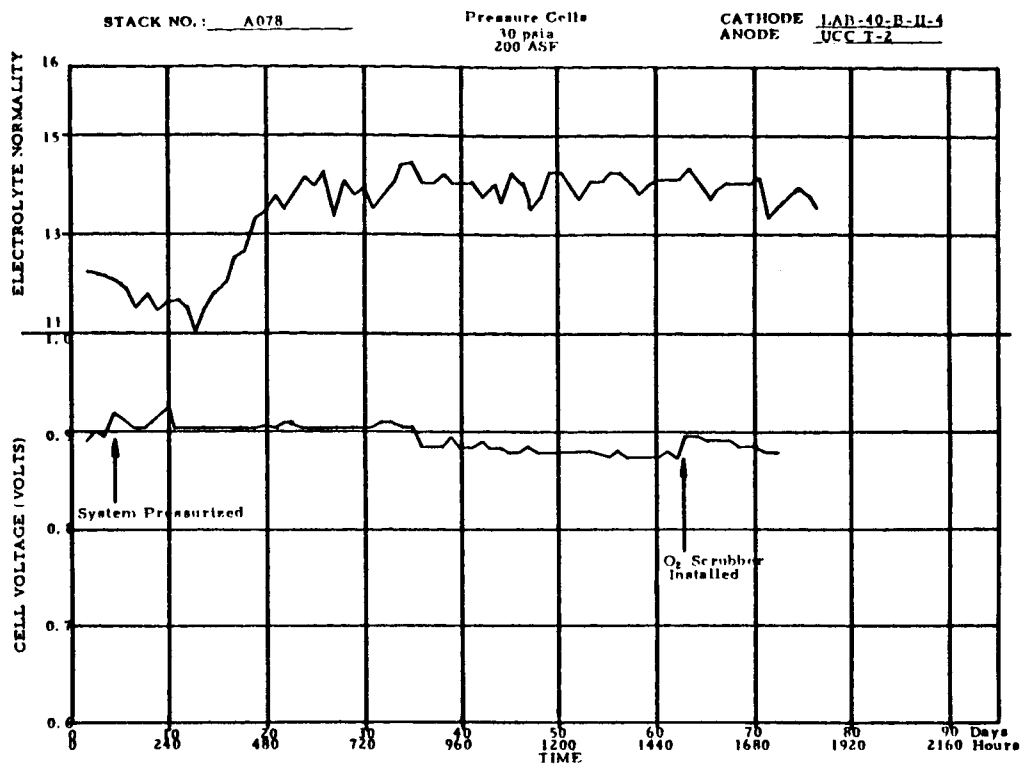D-3479

Fig. 26 - Time Versus Voltage - Stack A-055.

Fig. 27 - Time Versus Voltage - Stack A-059.

D-3480

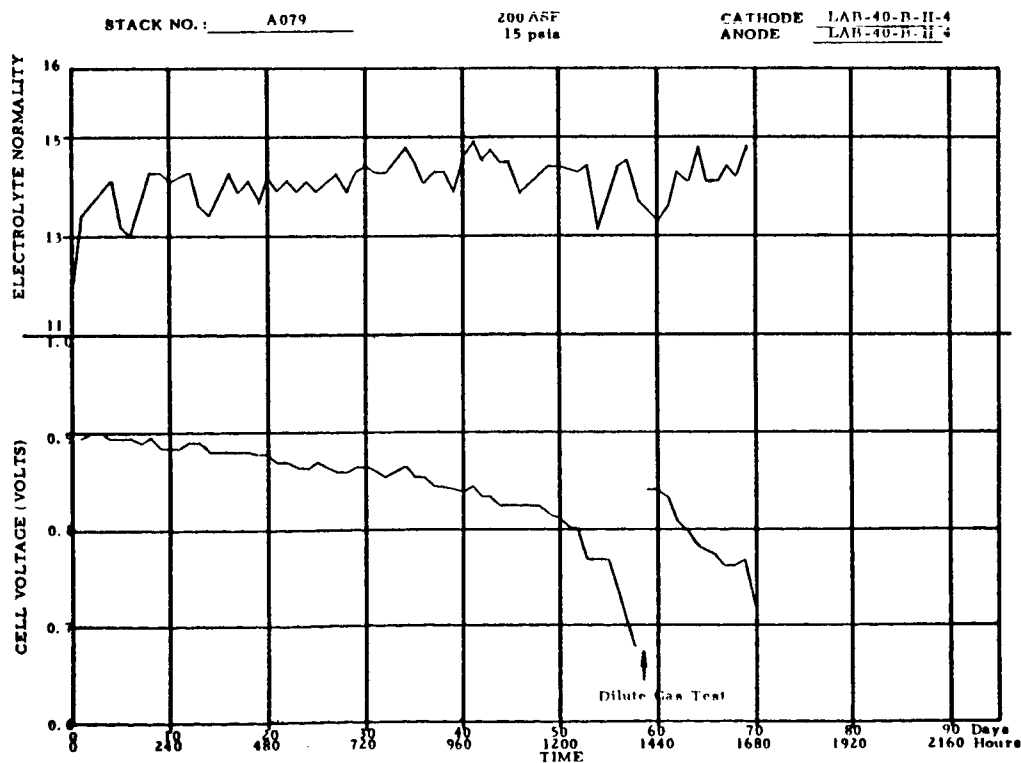**Fig. 28 - Time Versus Voltage - Stack A-060.**

D-3481

Fig. 29 - Time Versus Voltage - Stack A-061.

D-3482

Fig. 30 - Time Versus Voltage - Stack A-066.
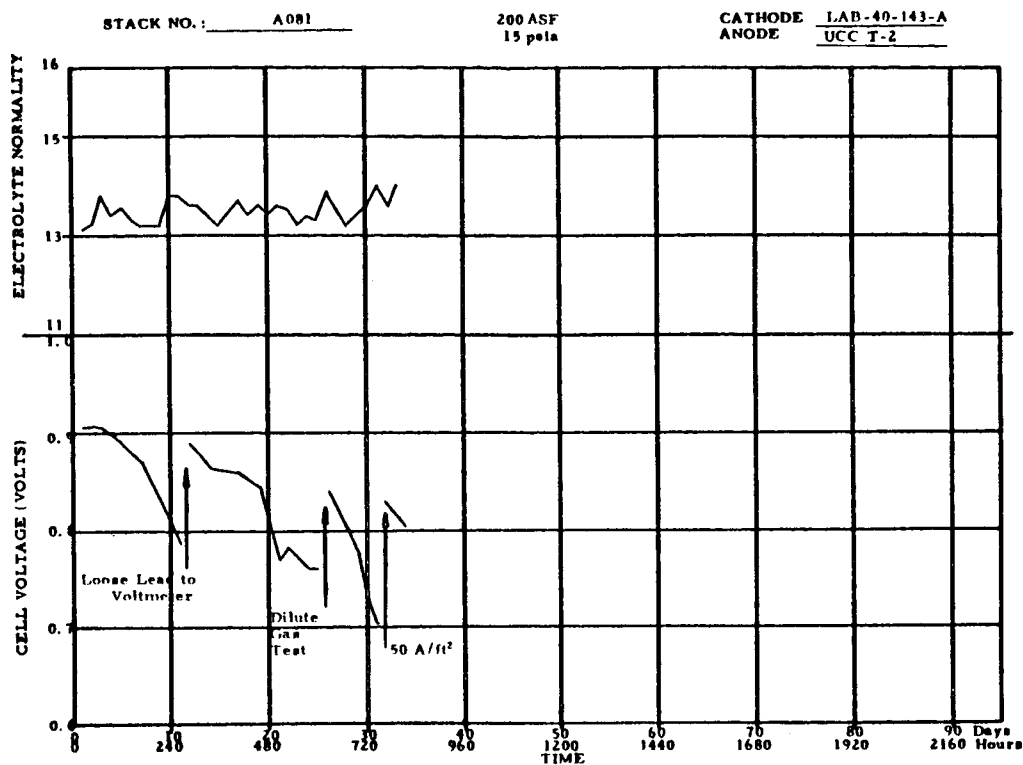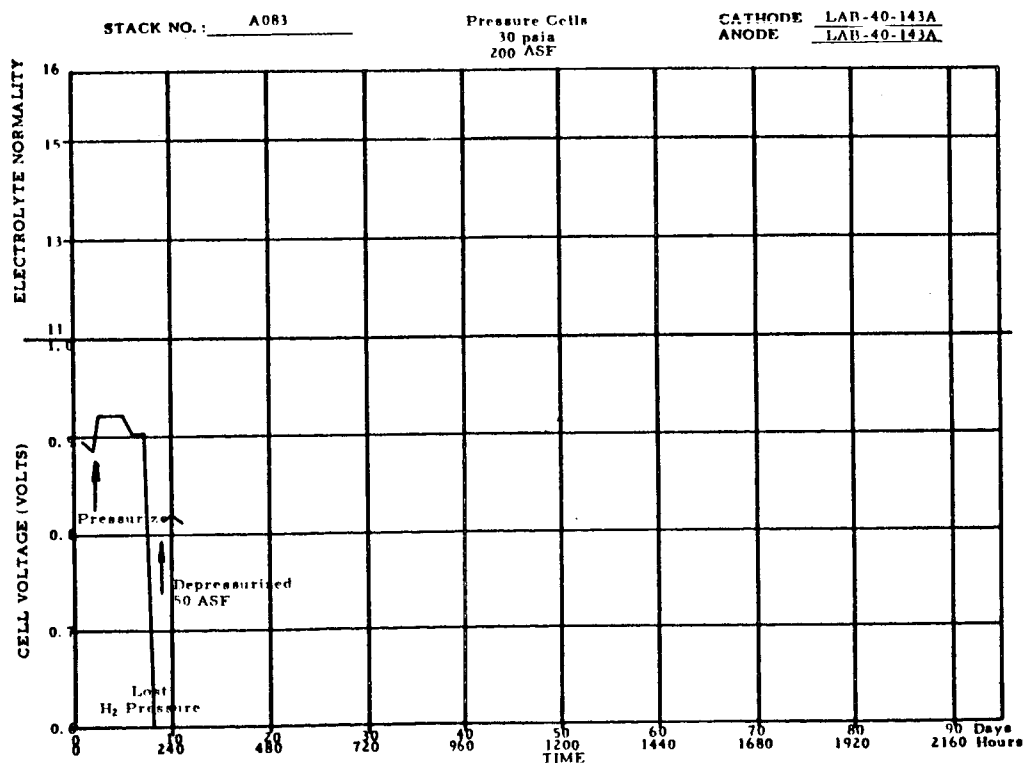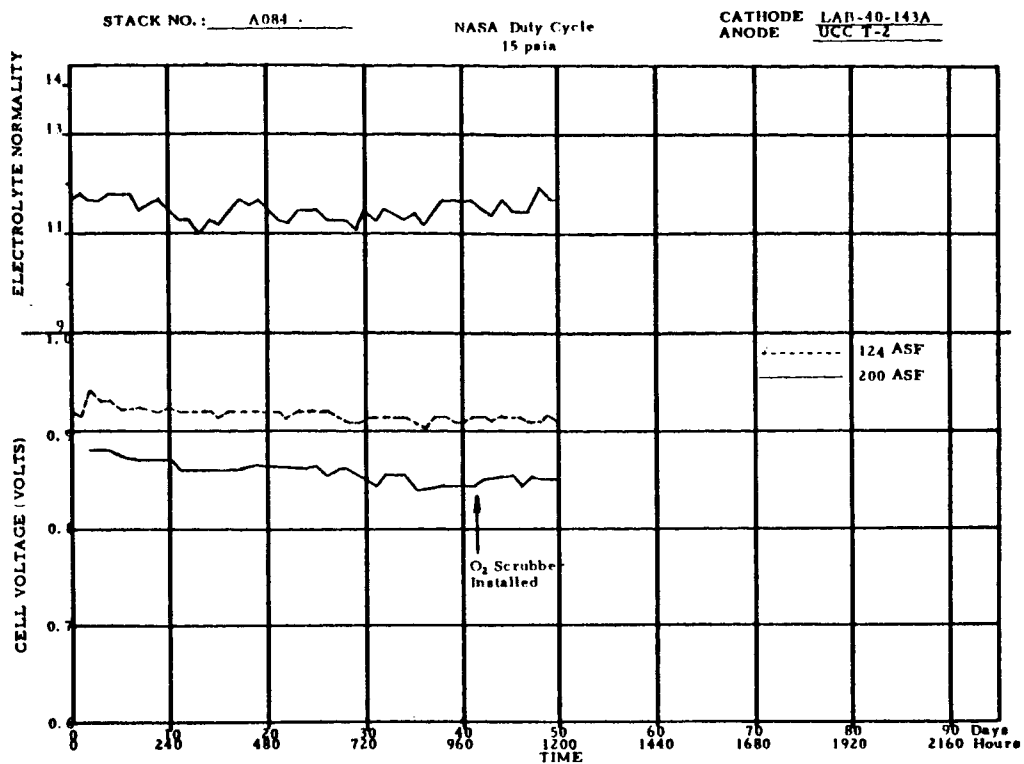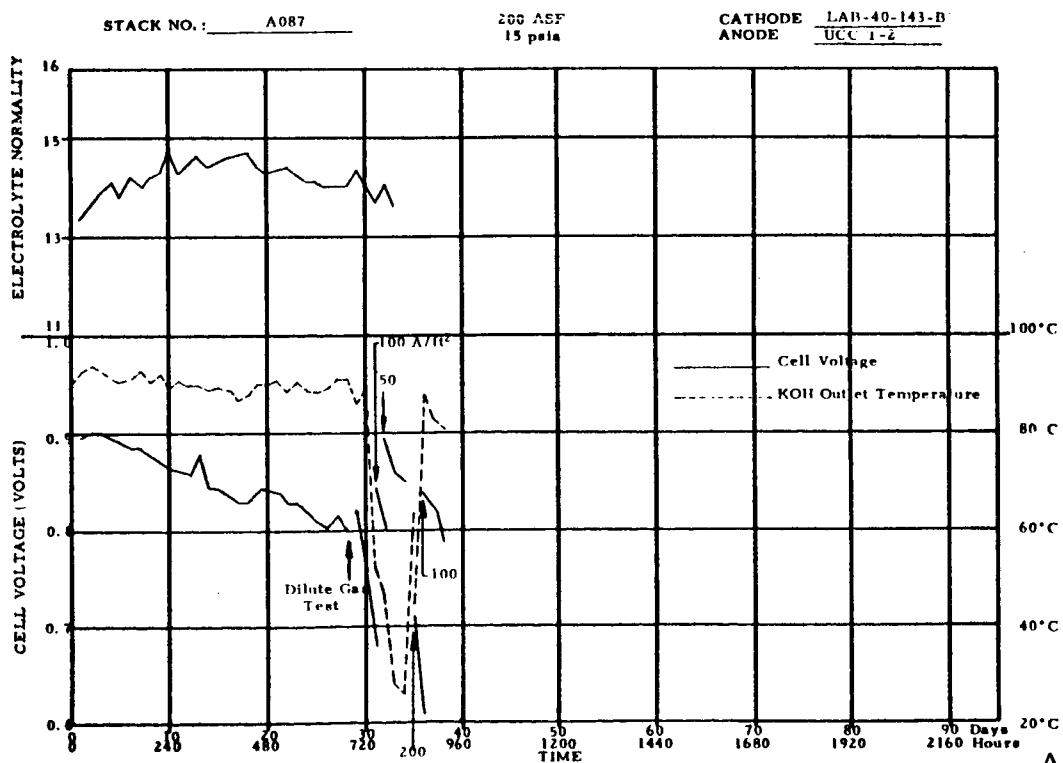
D-3483

STACK NO.: _____A068'_____   200 ASF   CATHODE LAB 40-A-II-2
                              15 psia   ANODE   LAB-40-A-II-2

Fig. 31 - Time Versus Voltage - Stack A-068.

D-3484

STACK NO.: _____A069_____   Navy Duty Cycle   CATHODE LAB-40-A-II-2
                             15 psia   ANODE   UCC-T-2

Fig. 32 - Time Versus Voltage - Stack A-069.

D-3485

-57-

STACK NO.: A071   200ASF   CATHODE LAB-40-A-II-1
           15 psia   ANODE UCC T-2

Fig. 33 - Time Versus Voltage - Stack A 071.

D-3486

STACK NO.: A072   300 ASF   CATHODE LAB-40-A-II-1
           15 psia   ANODE UCC T-2

200 A/ft²

Fig. 34 - Time Versus Voltage - Stack A-072.

D-3487

-58-

STACK NO.: A073   NASA Duty Cycle 15 psia   CATHODE LAB-40-A-II-2 ANODE UCC T-2

ELECTROLYTE NORMALITY

CELL VOLTAGE (VOLTS)

124 ASF
200 ASF

TIME

D-3488

Fig. 35 - Time Versus Voltage - Stack A-073.

STACK NO.: A074   Pressure Cells 30 psia 200 ASF   CATHODE LAB-40-II-1 ANODE UCC T-2

ELECTROLYTE NORMALITY

CELL VOLTAGE (VOLTS)

Pressurized

Removed from Pressure Stand

TIME

D-3489

Fig. 36 - Time Versus Voltage - Stack A-074.

-59-

Fig. 37 - Time Versus Voltage - Stack A-075.

D-3490

D-3491

Fig. 38 - Time Versus Voltage - Stack A-077.

Fig. 39 - Time Versus Voltage - Stack A-078.

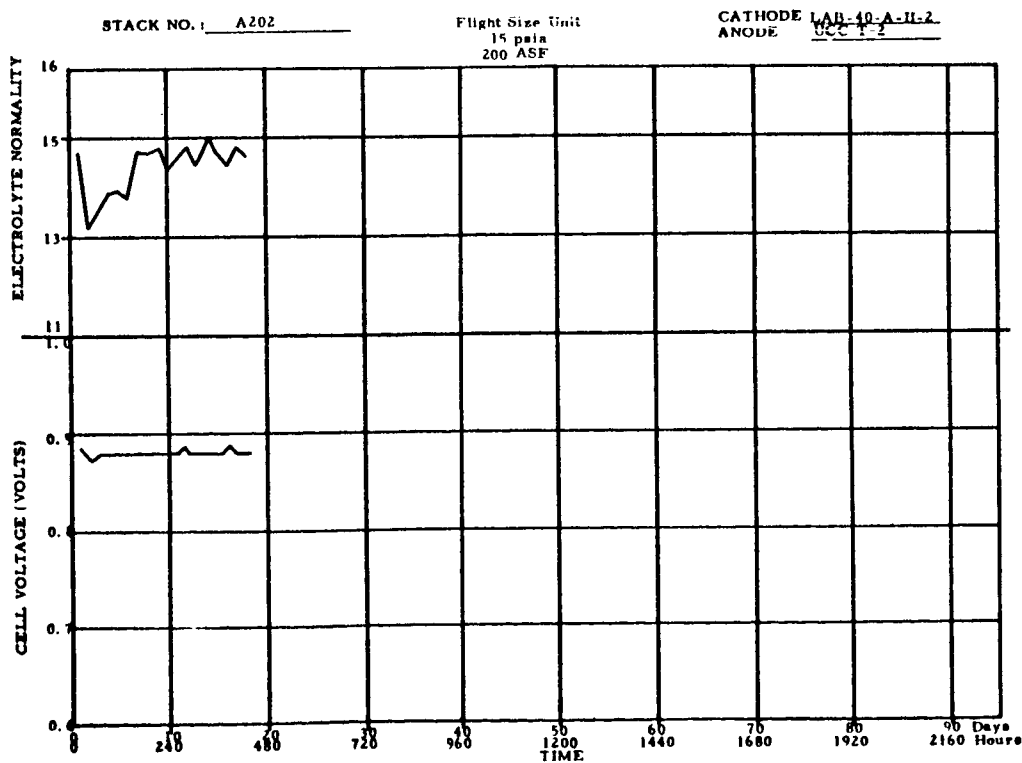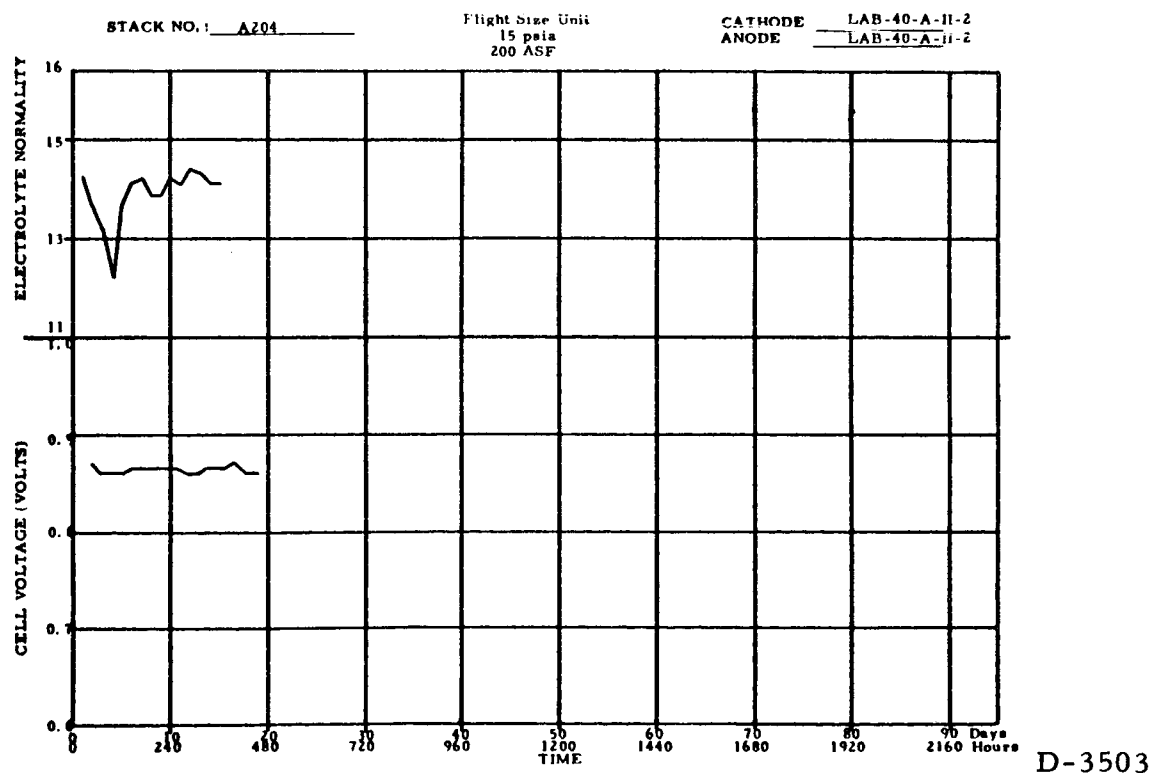D-3492

Fig. 40 - Time Versus Voltage - Stack A-079.

D-3493
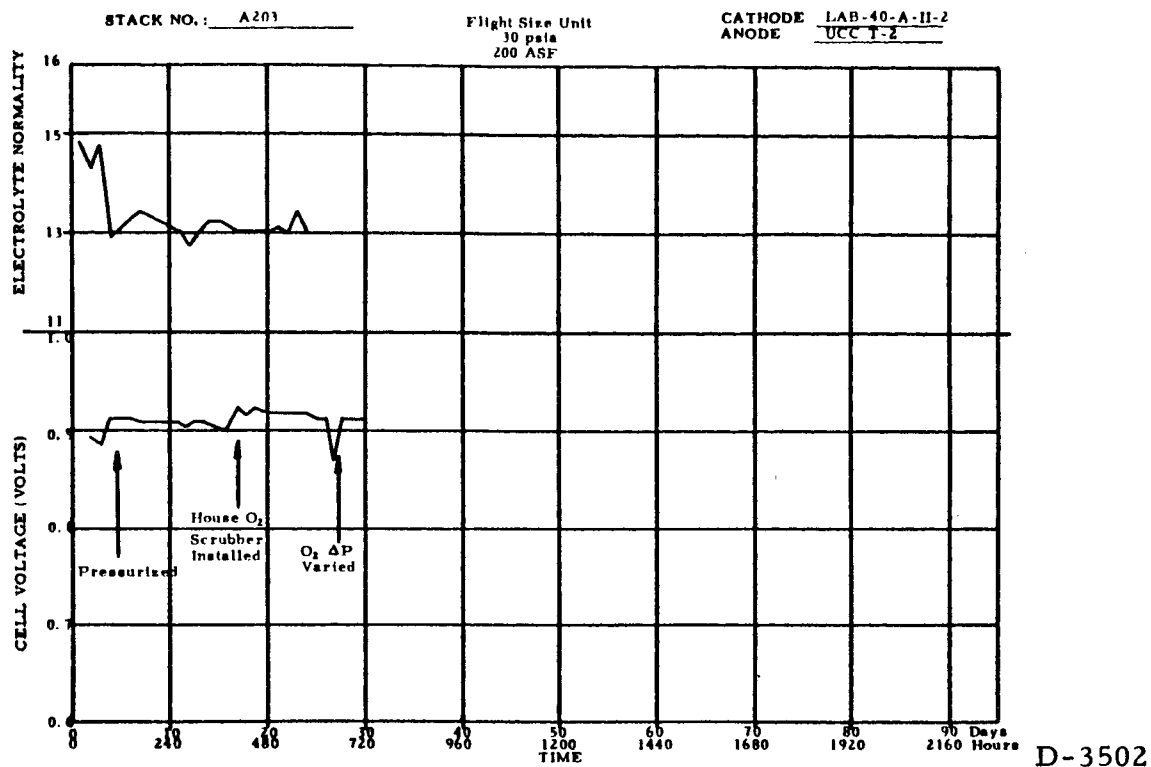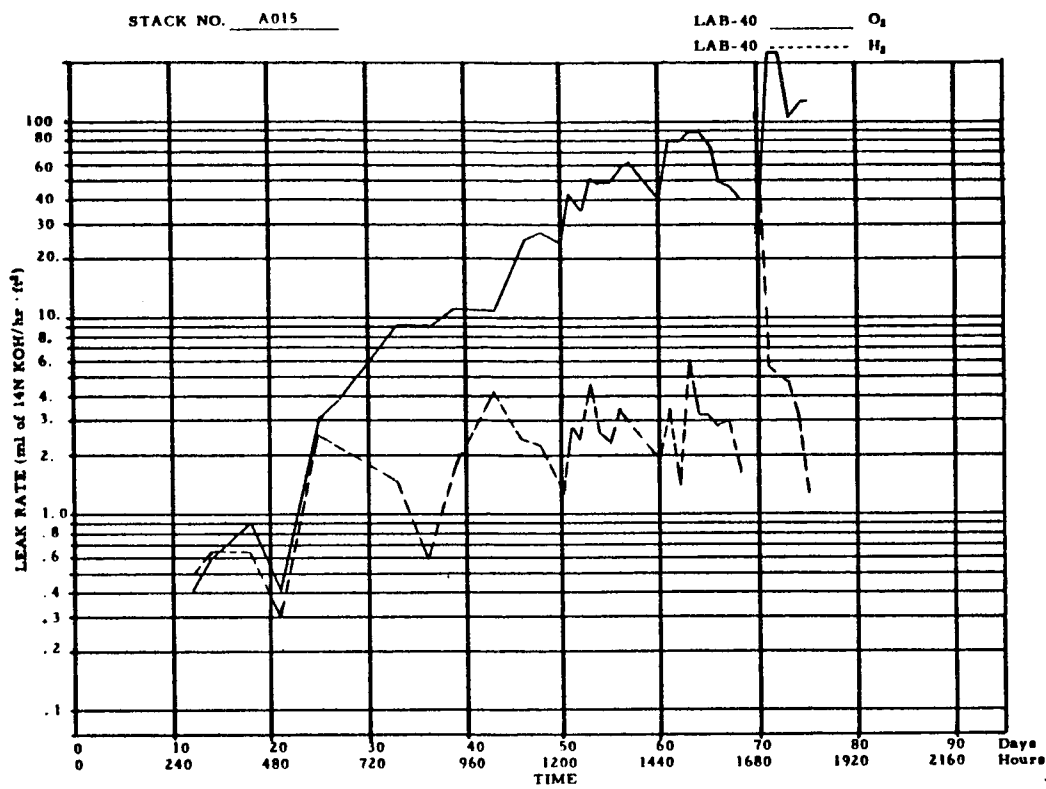
STACK NO.: A081   200 ASF   CATHODE LAB-40-143-A
15 psia   ANODE UCC T-2

D-3494

Fig. 41 - Time Versus Voltage - Stack A-081.

STACK NO.: A083   Pressure Cells   CATHODE LAB-40-143A
30 psia   ANODE LAB-40-143A
200 ASF

D-3495

Fig. 42 - Time Versus Voltage - Stack A-083.
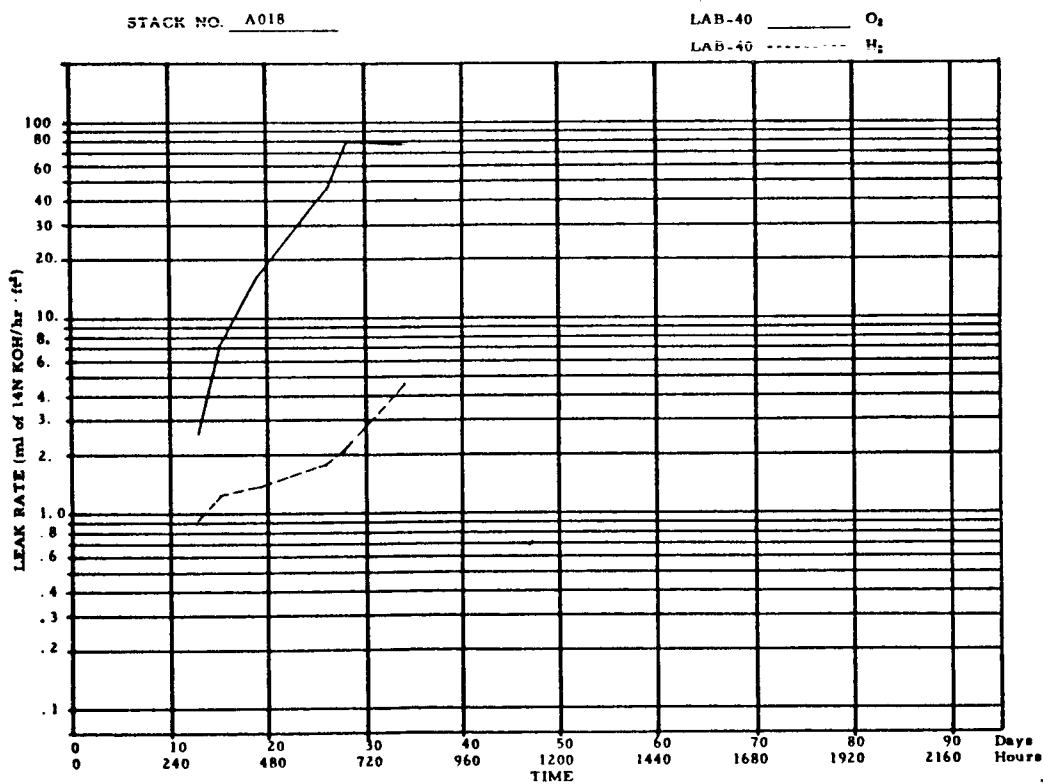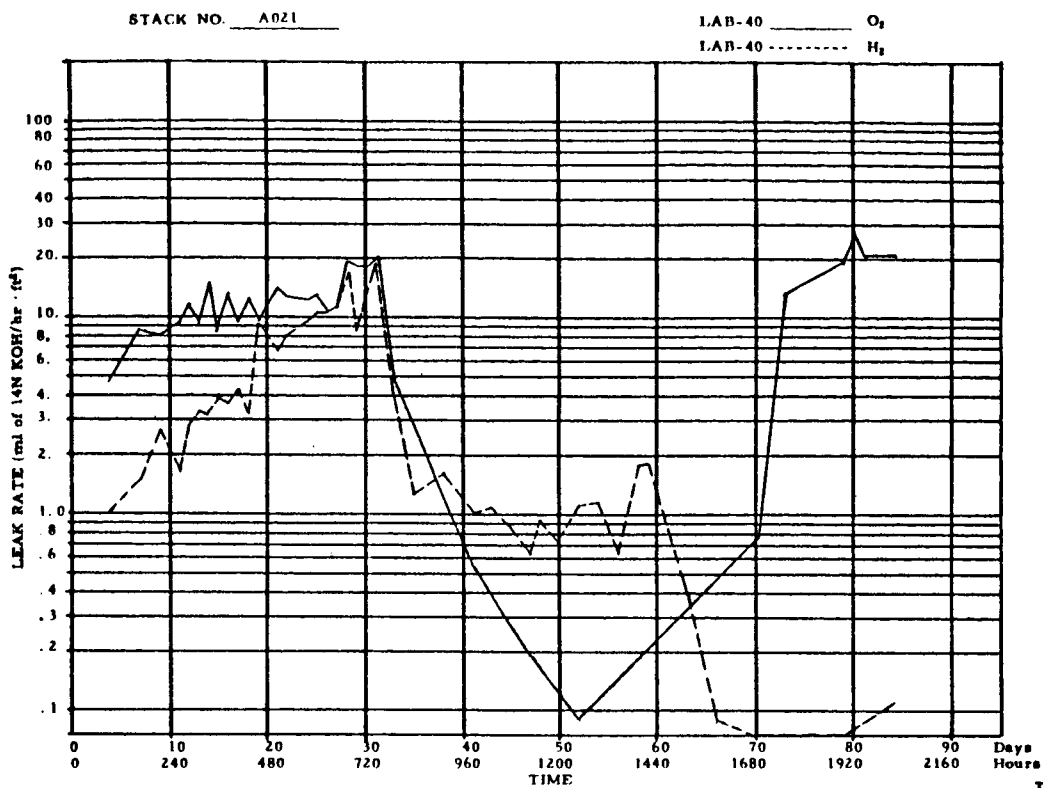
-62-

Fig. 43 - Time Versus Voltage - Stack A-084.

Fig. 44 - Time Versus Voltage - Stack A-087.

Pressure Cells
30 psia
200 ASF

CATHODE LAB-40-143B
ANODE LAB-40-143B

ELECTROLYTE NORMALITY

CELL VOLTAGE (VOLTS)

Pressurize

TIME

D-3498

Fig. 45 - Time Versus Voltage - Stack A-089.

STACK NO. : ___A090___

NASA Duty Cycle
15 psia

CATHODE LAB-40-143B
ANODE UCC T-2

ELECTROLYTE NORMALITY

CELL VOLTAGE (VOLTS)

·········· 124 ASF
———— 200 ASF

O₂ Scrubber
Installed

TIME

D-3499

Fig. 46 - Time Versus Voltage - Stack A-090.

-64-
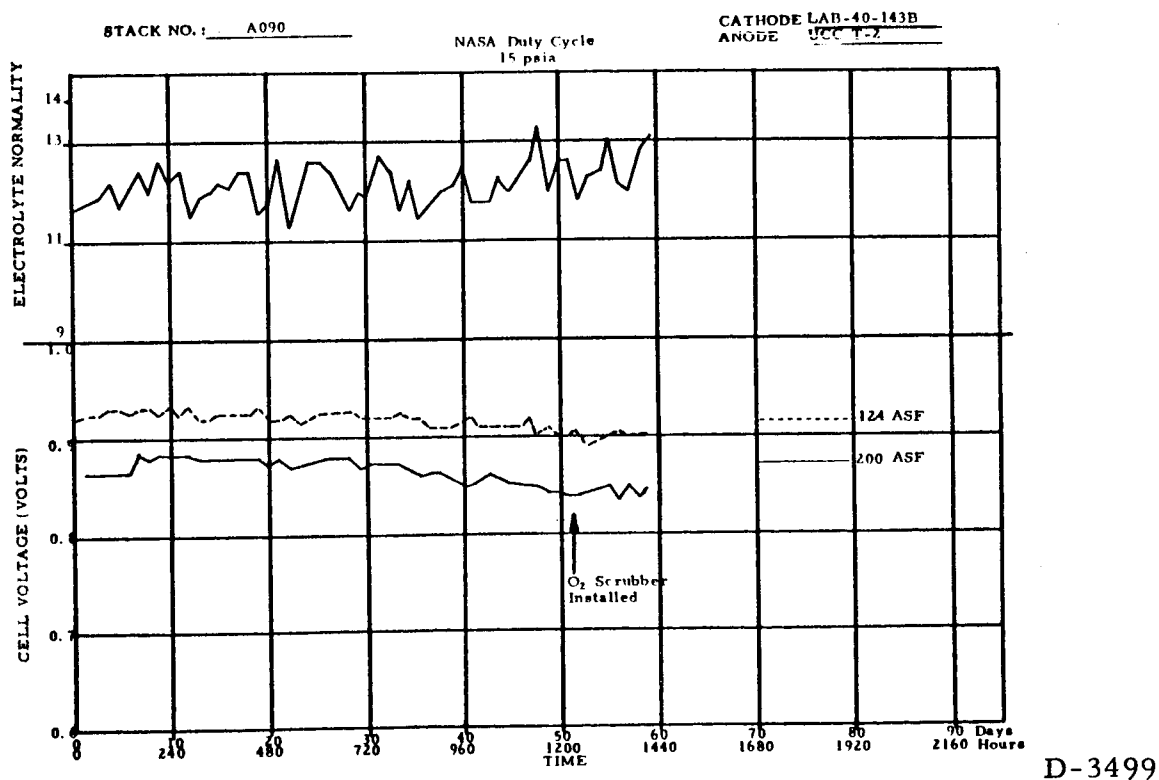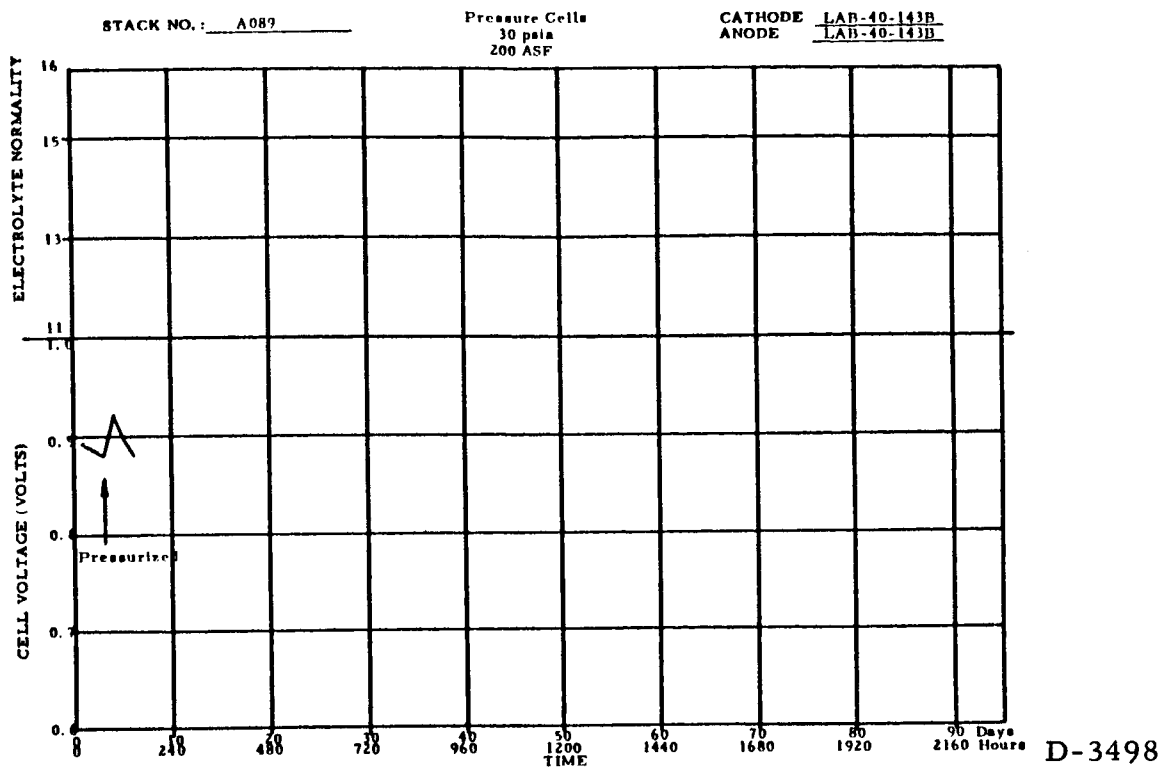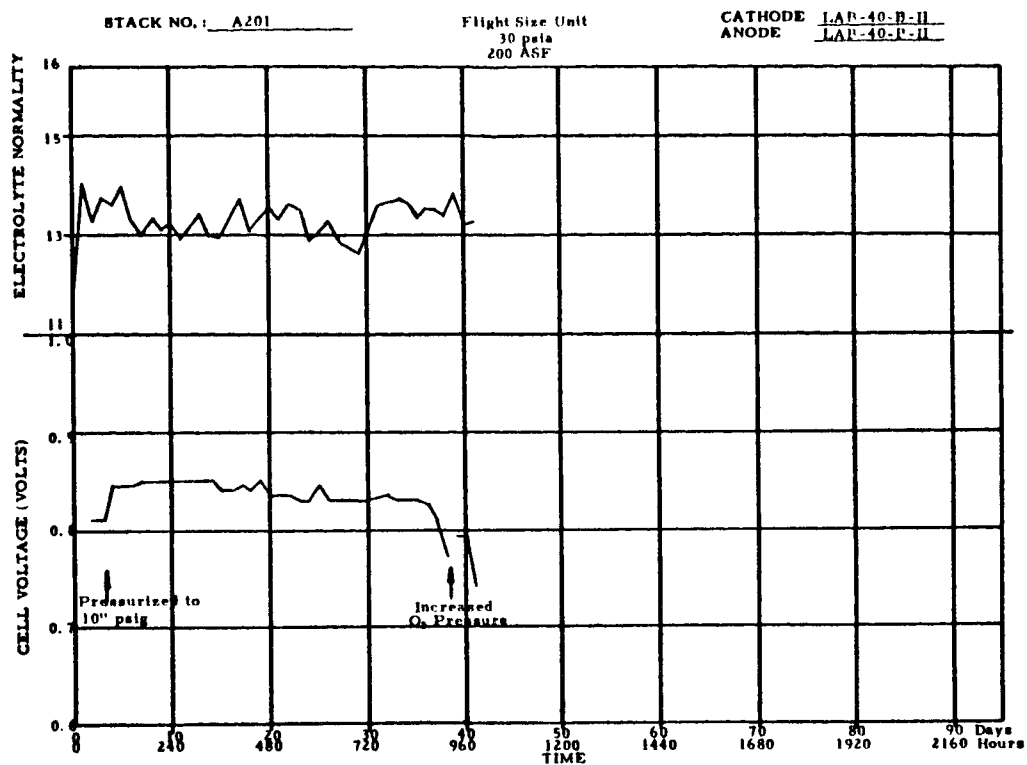
Fig. 47 - Time Versus Voltage - Stack A-201.

Fig. 48 - Time Versus Voltage - Stack A-202.

-65-

Fig. 49 - Time Versus Voltage - Stack A-203.

Fig. 50 - Time Versus Voltage - Stack A-204.

-66-

STACK NO. A015 LAB-40 ———— O₂ LAB-40 ----- H₂

D-3504

Fig. 51 - Leak Rate - Stack A-015.

STACK NO. A018 LAB-40 ———— O₂ LAB-40 ----- H₂

D-3505

Fig. 52 - Leak Rate - Stack A-018.

STACK NO. A021   LAB-40 ———— O₂
                   LAB-40 -------- H₂

D-3506

Fig. 53 - Leak Rate - Stack A-021.

STACK NO. A022   LAB-40 ———— O₂
                   LAB-40 -------- H₂
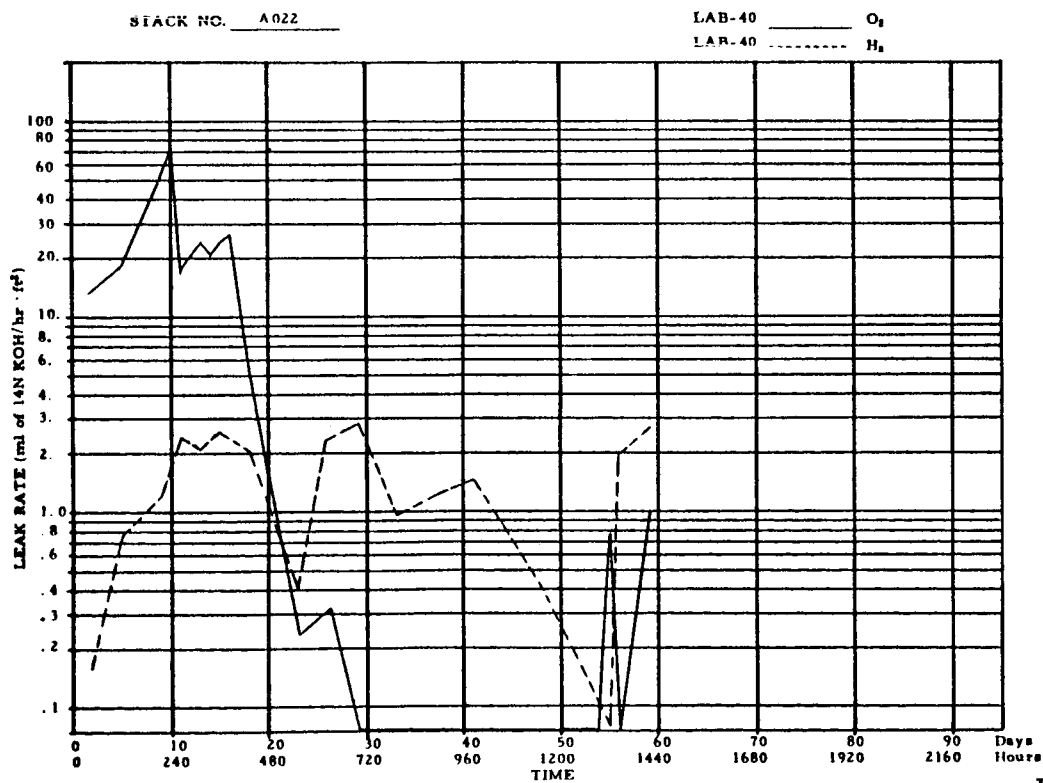
D-3507

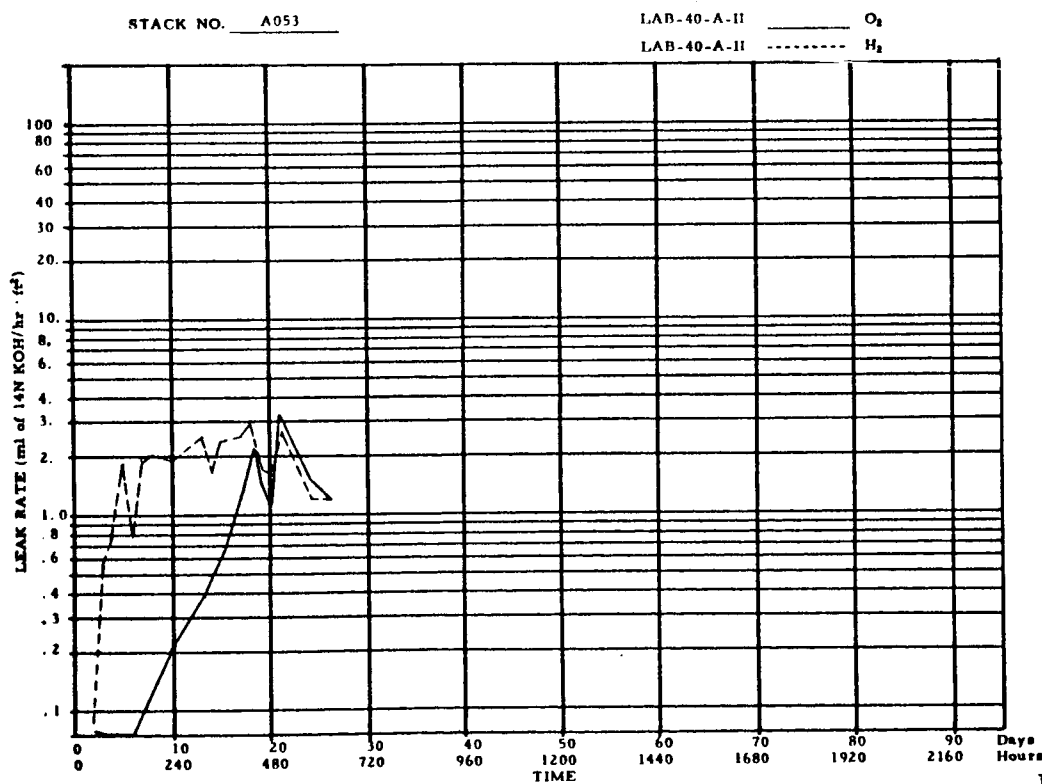Fig. 54 - Leak Rate - Stack A-022.
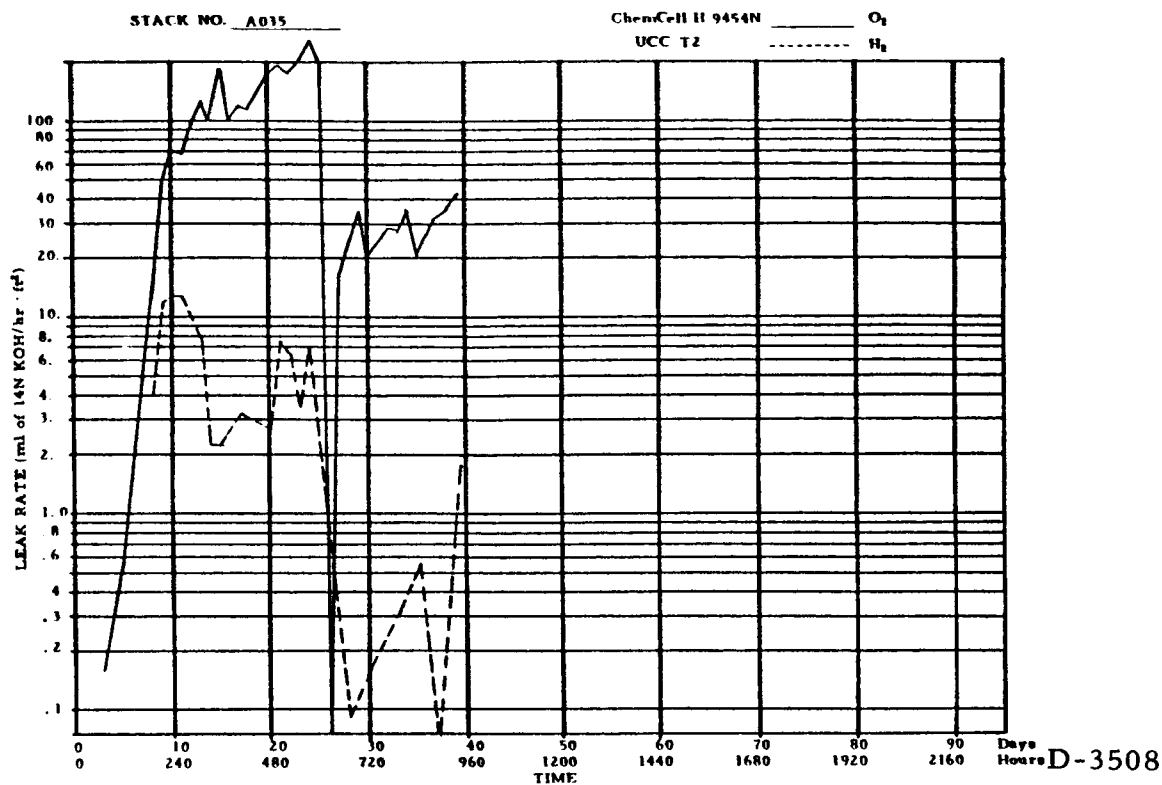
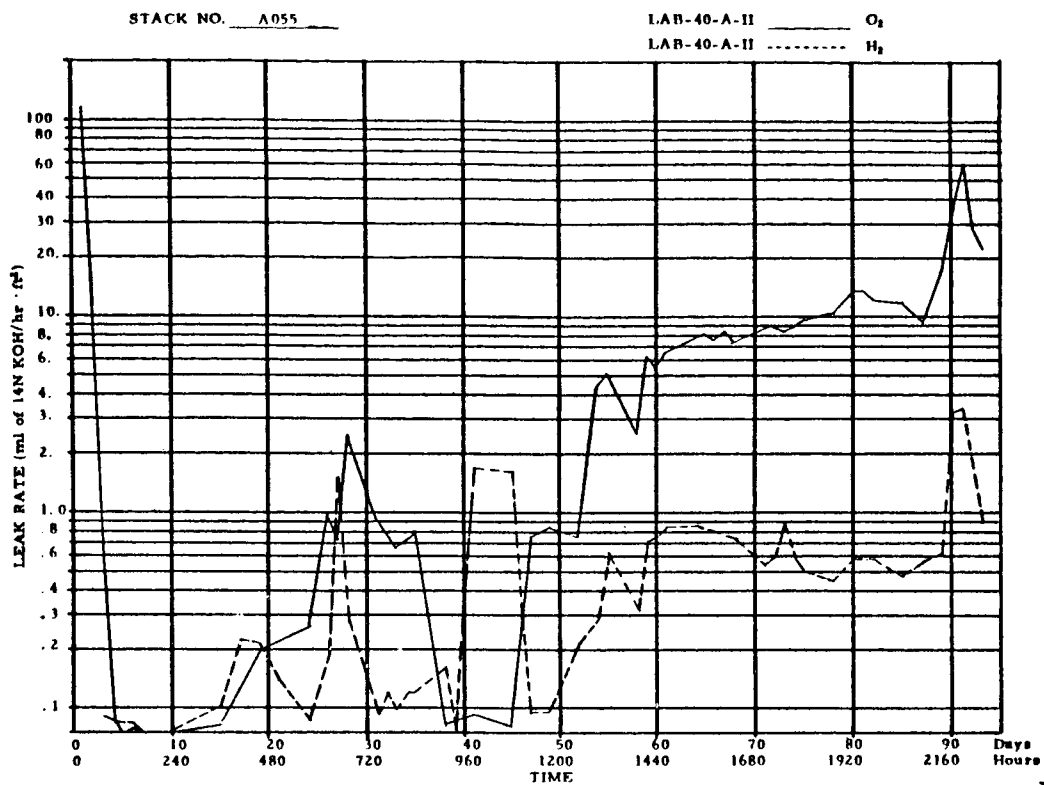Fig. 55 - Leak Rate - Stack A-035.
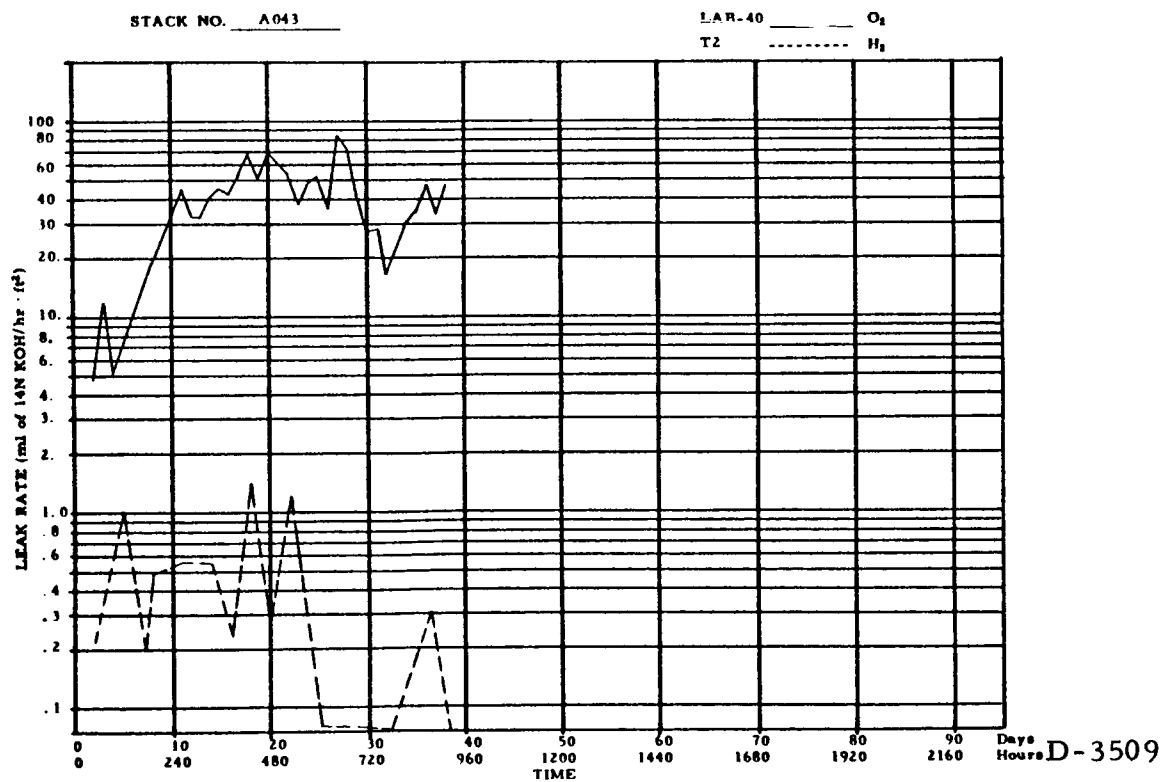
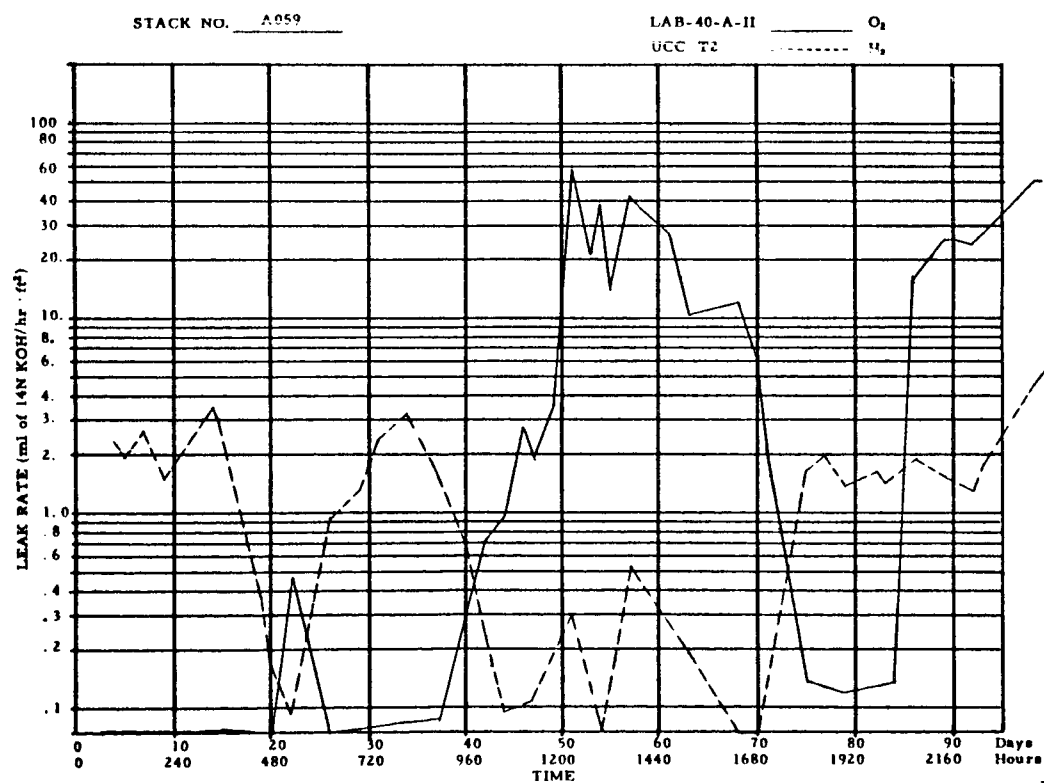Fig. 56 - Leak Rate - Stack A-043.
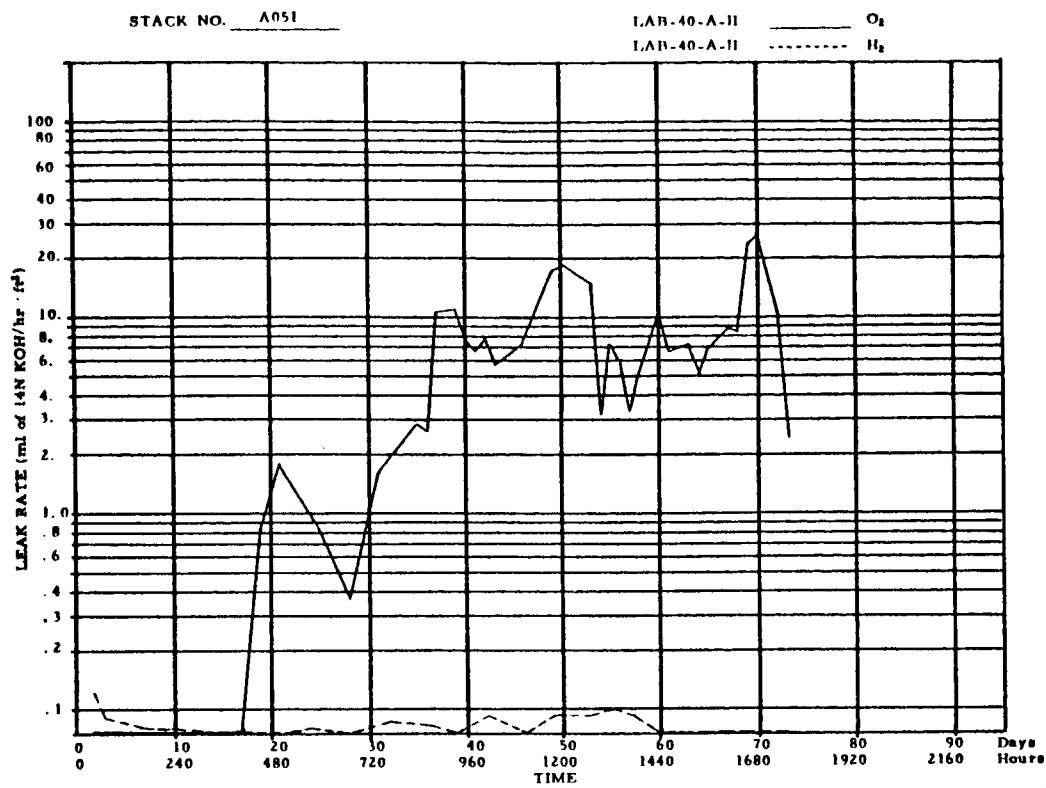
Fig. 57 - Leak Rate - Stack A-051.

D-3510

Fig. 58 - Leak Rate - Stack A-053.

D-3511

-70-

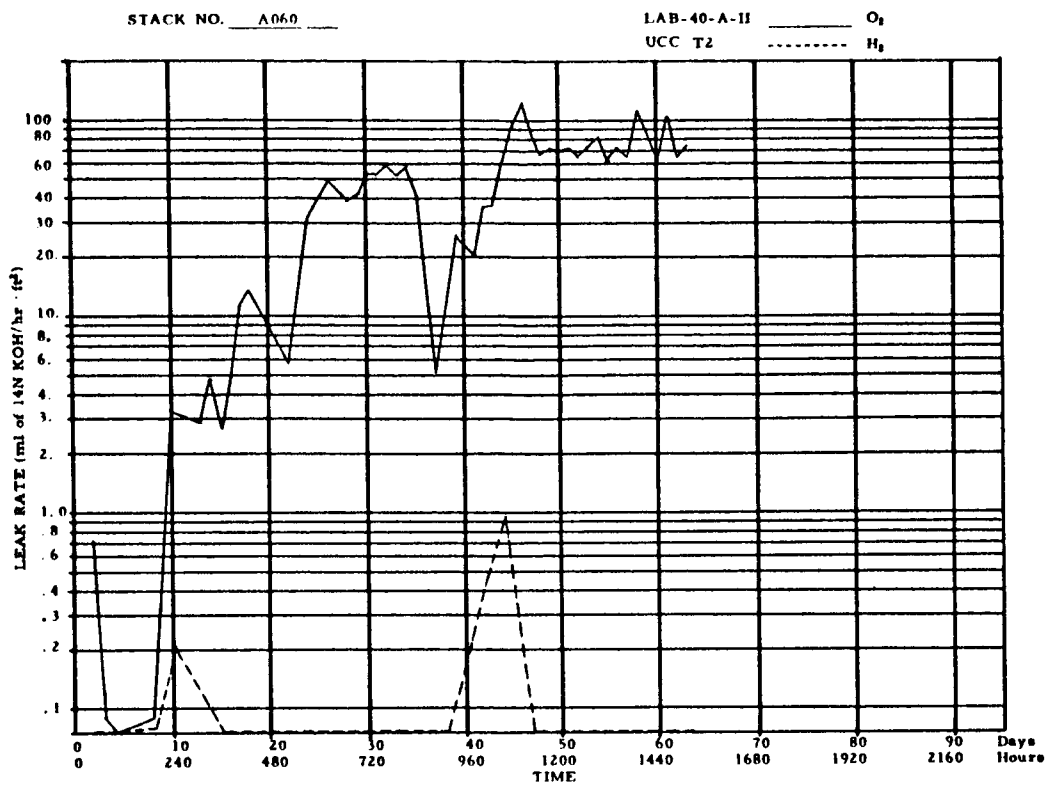D-3512

Fig. 59 - Leak Rate - Stack A-055.
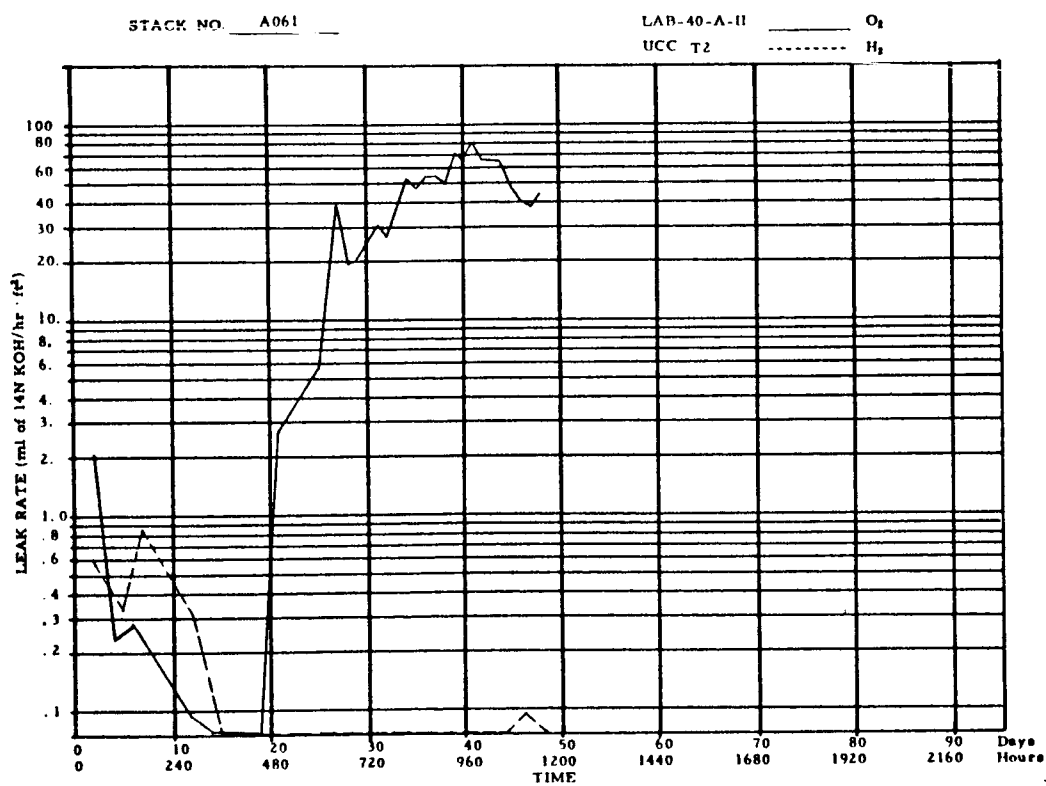
D-3513

Fig. 60 - Leak Rate - Stack A-059.

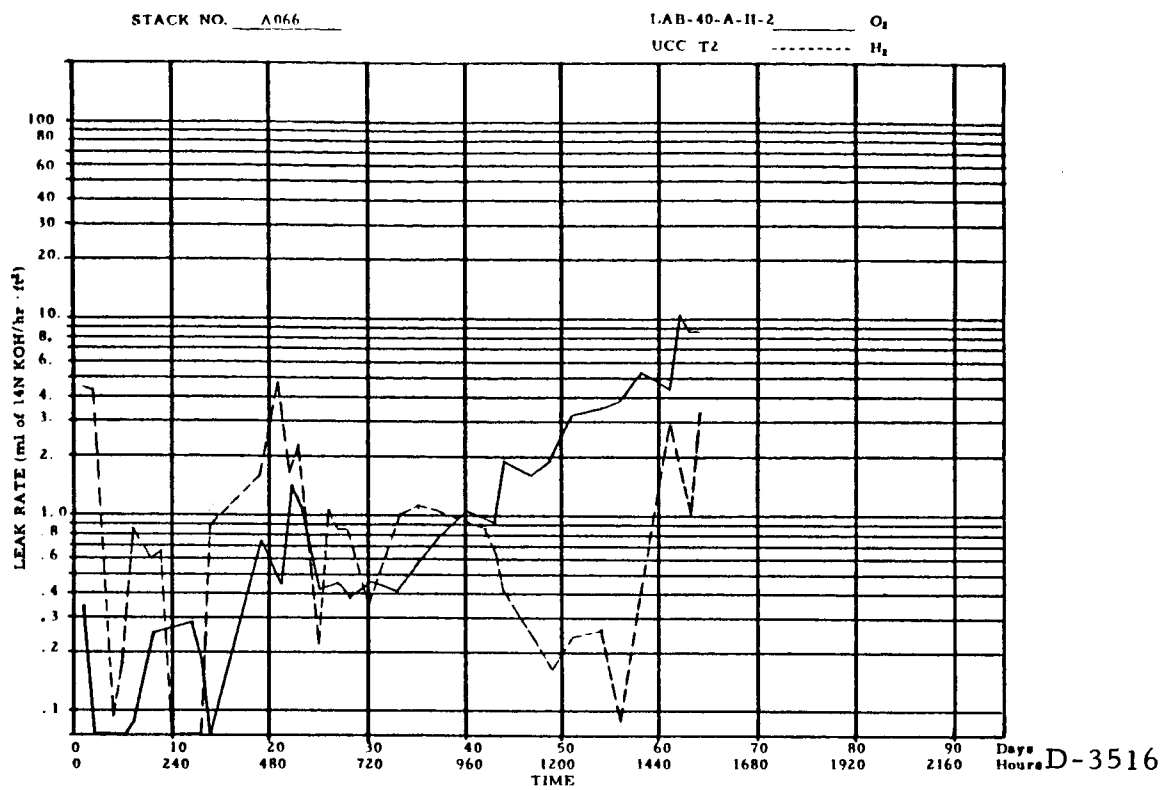Fig. 61 - Leak Rate - Stack A-060.

Fig. 62 - Leak Rate - Stack A-061.

Fig. 63 - Leak Rate - A-066.

D-3516

Fig. 64 - Leak Rate - Stack A-068.

D-3517

-73-

Fig. 65 - Leak Rate - Stack A-069.

D-3518

Fig. 66 - Leak Rate - Stack A-071.

D-3519

D-3520

Fig. 67 - Leak Rate - Stack A-072.

D-3521

Fig. 68 - Leak Rate - Stack A-073.

-75-

Fig. 69 - Leak Rate - Stack A-074.
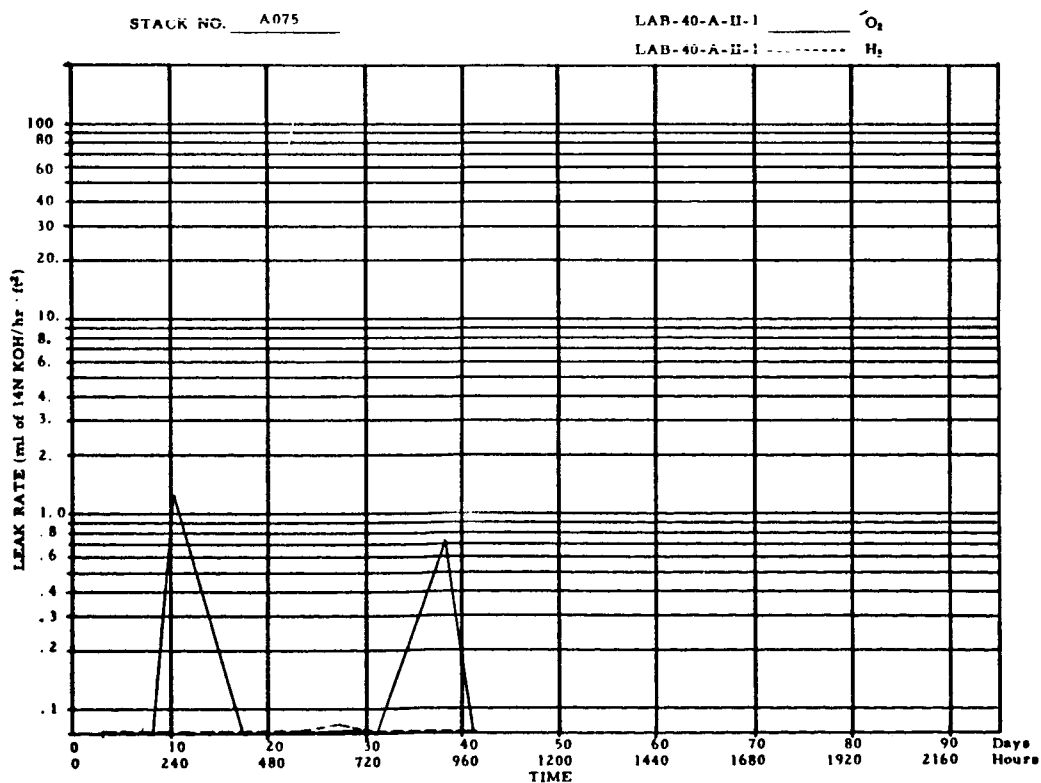
Fig. 70 - Leak Rate - Stack A-075.
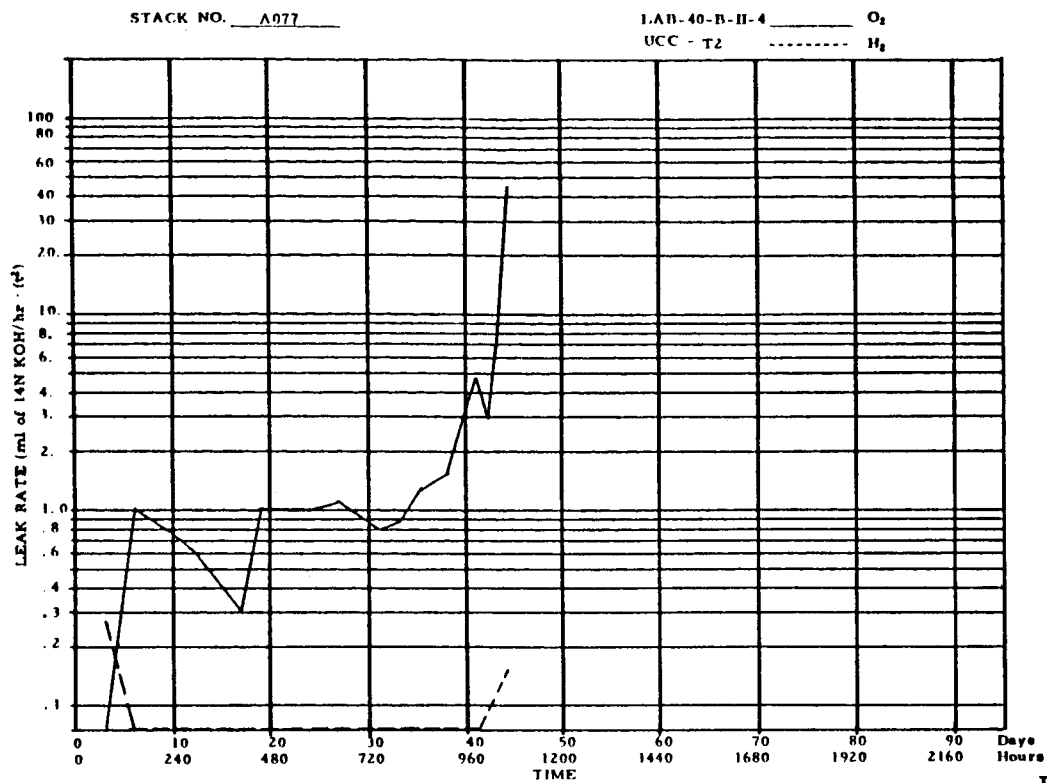
D-3524

Fig. 71 - Leak Rate - Stack A-077.
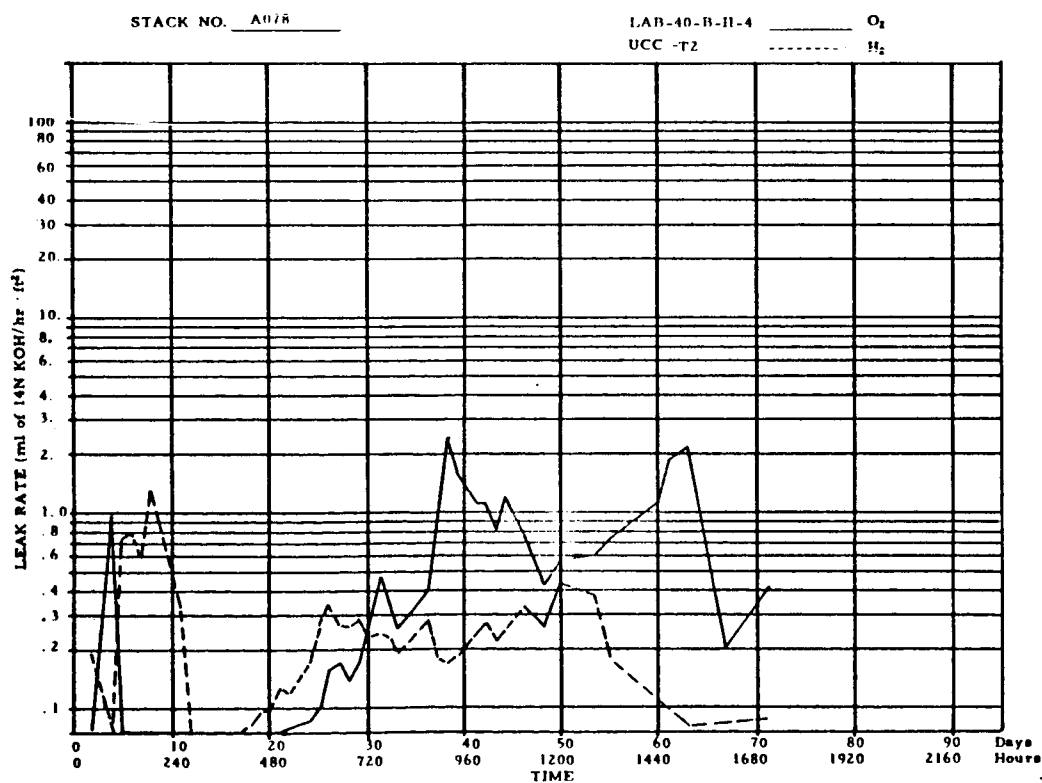
D-3525

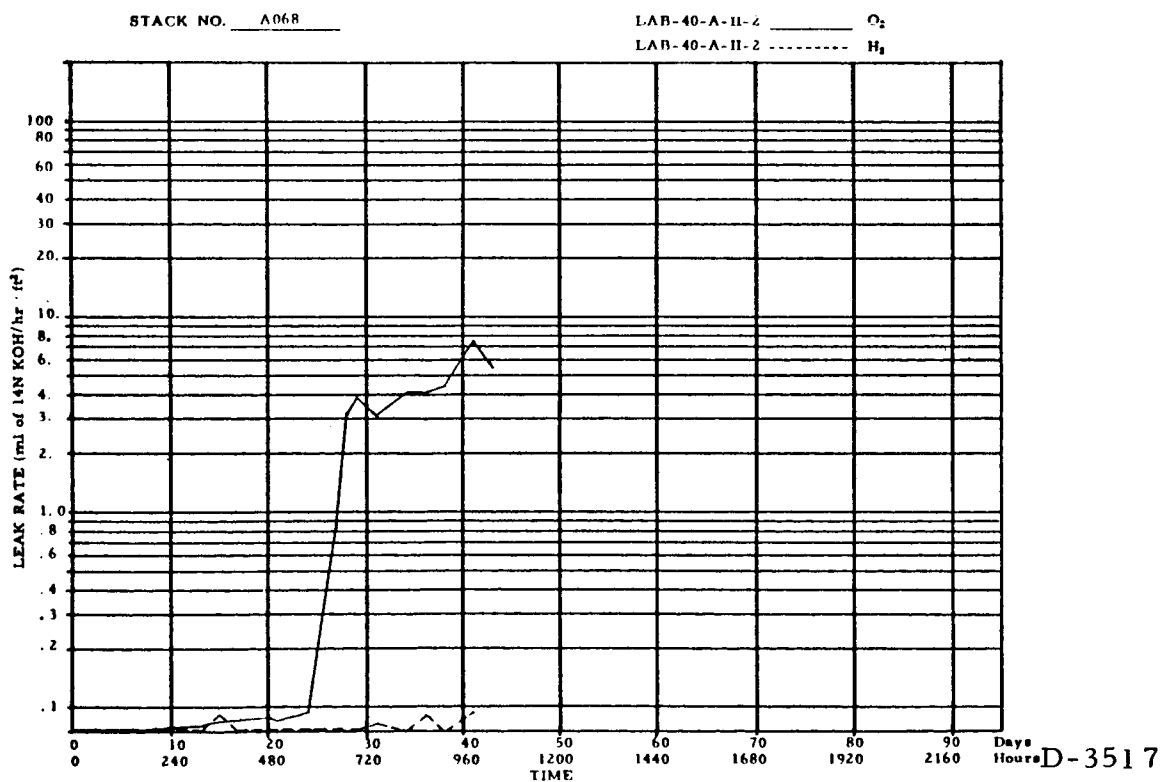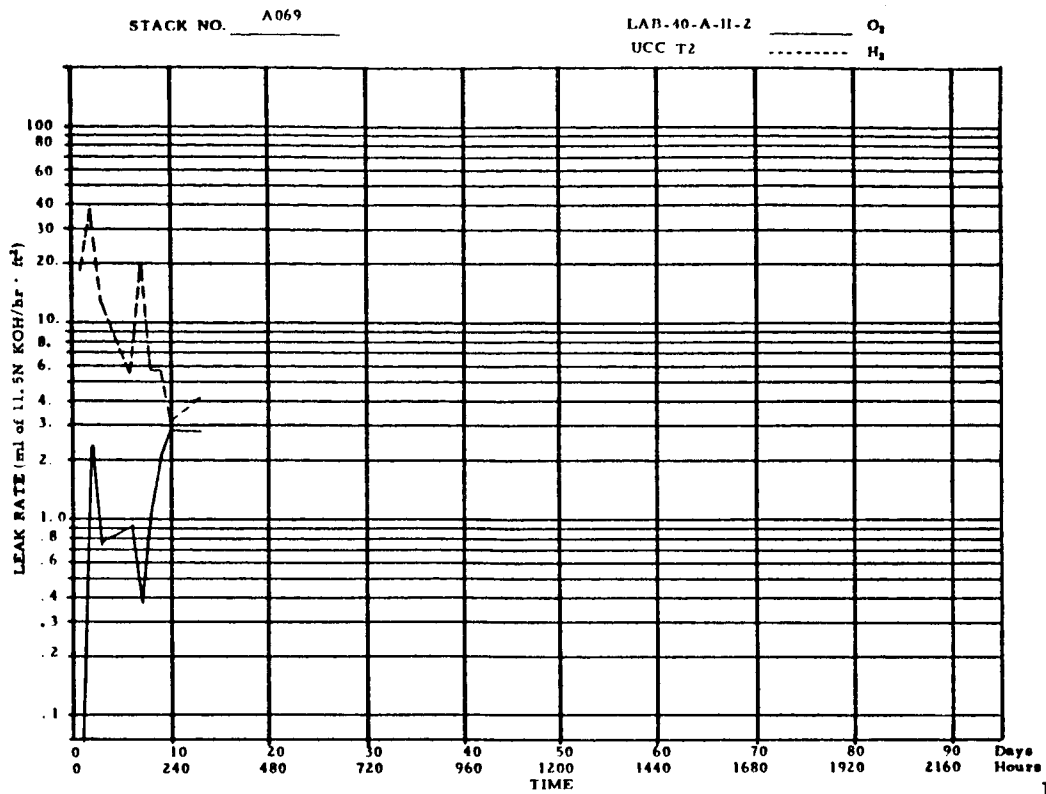Fig. 72 - Leak Rate - Stack A-078.
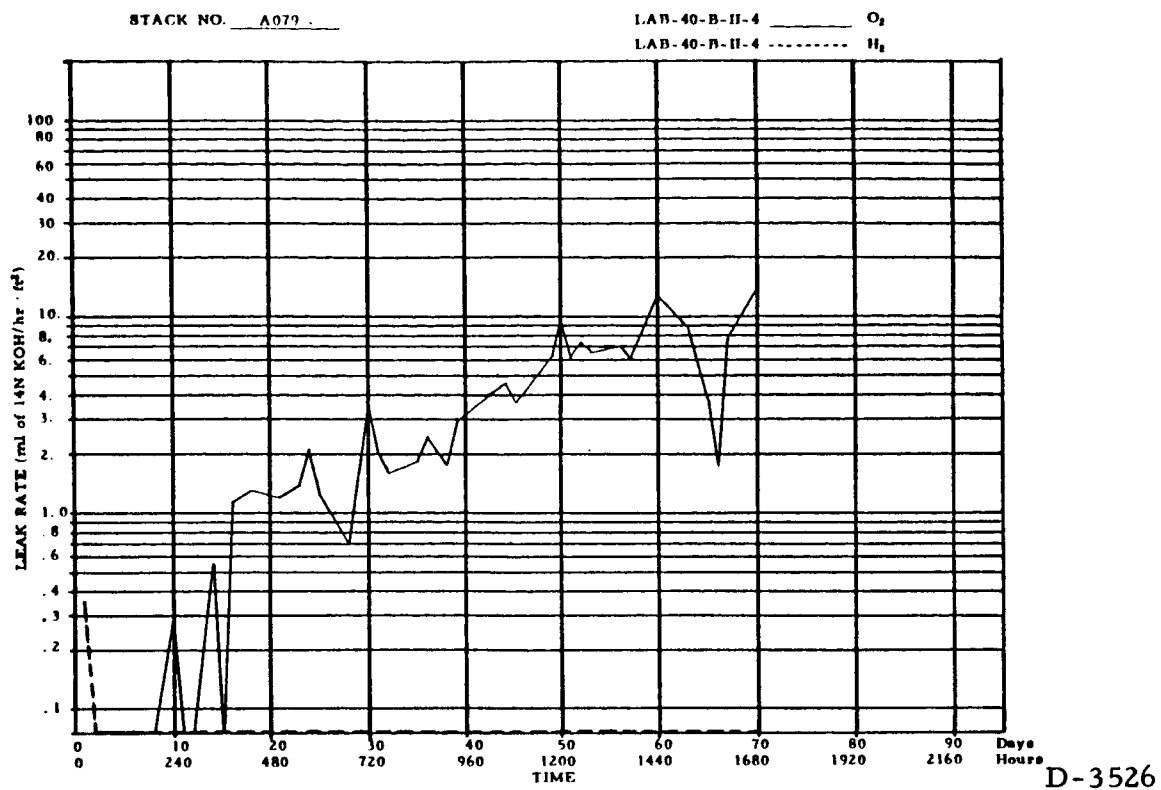
Fig. 73 - Leak Rate - Stack A-079.

D-3526
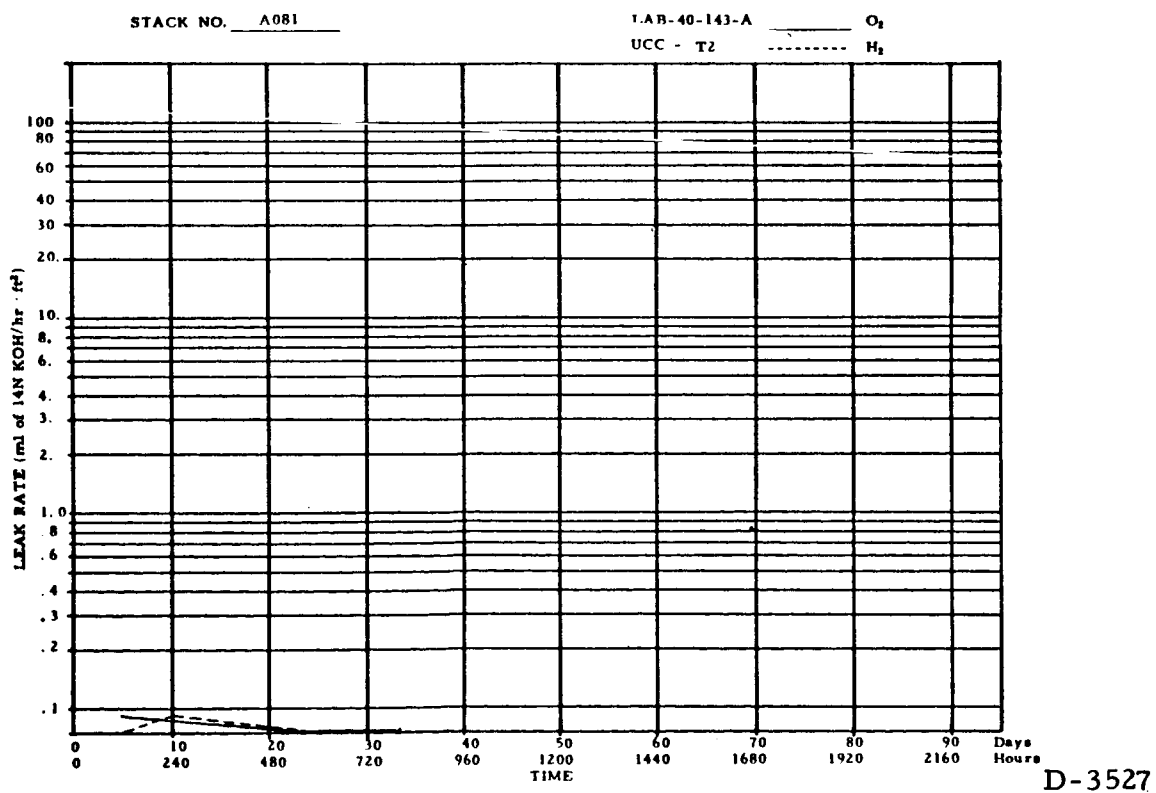
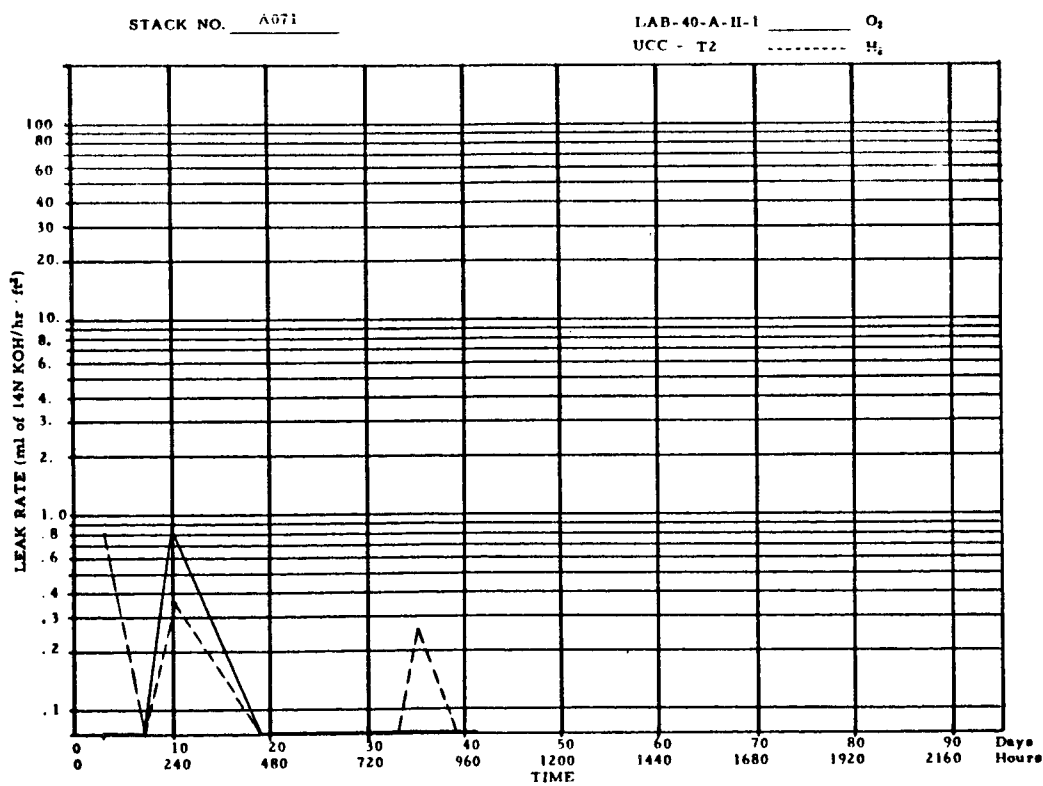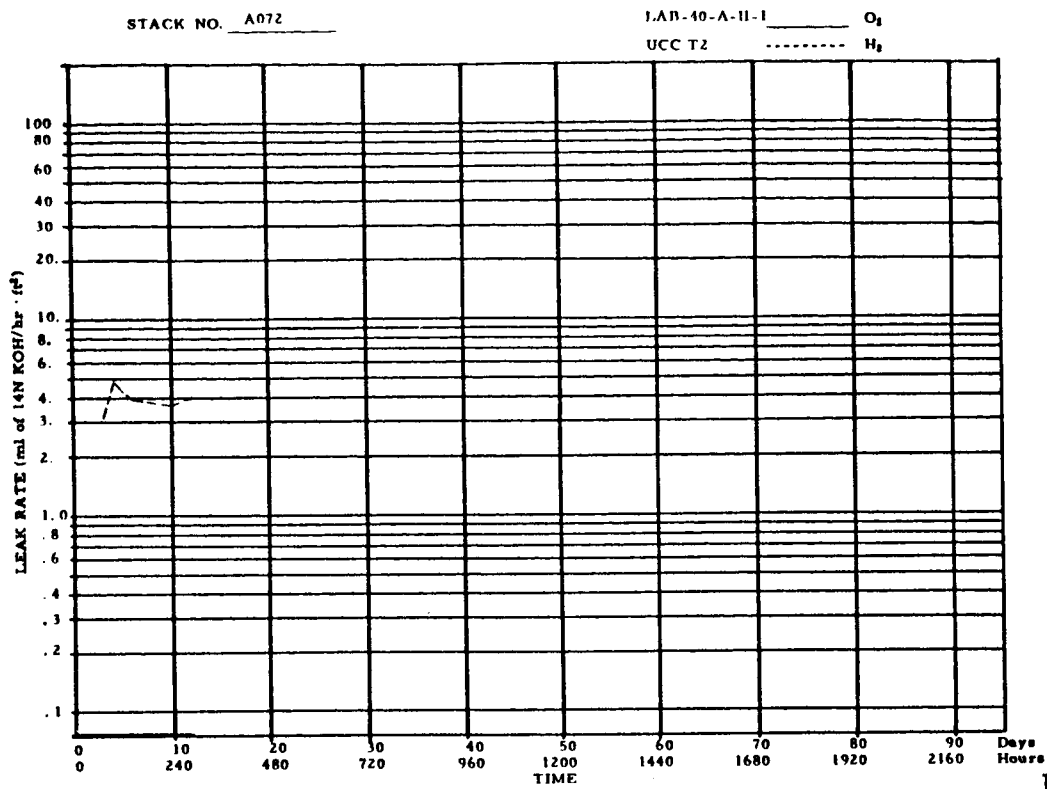Fig. 74 - Leak Rate - Stack A-081.

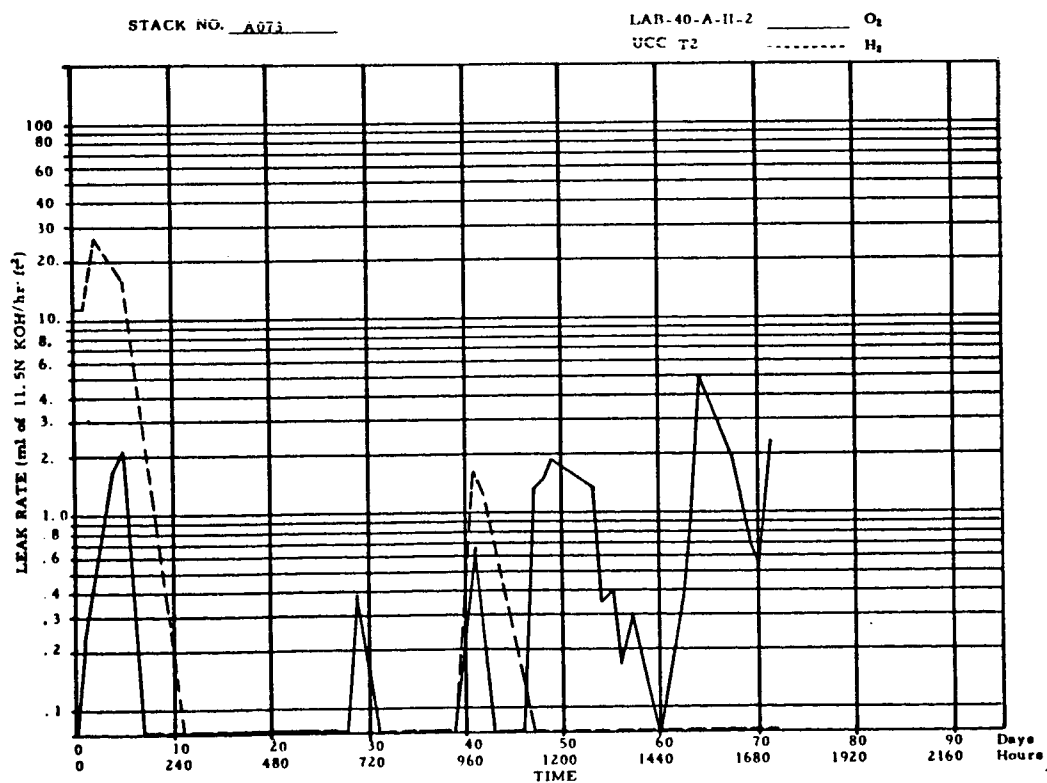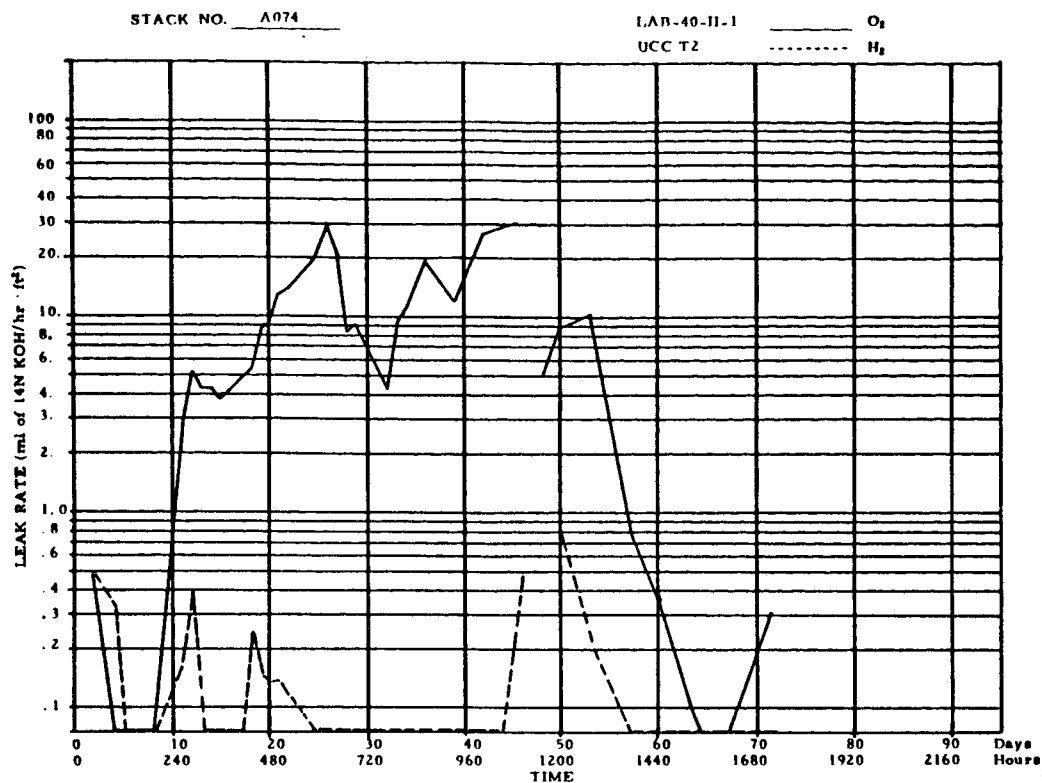D-3527
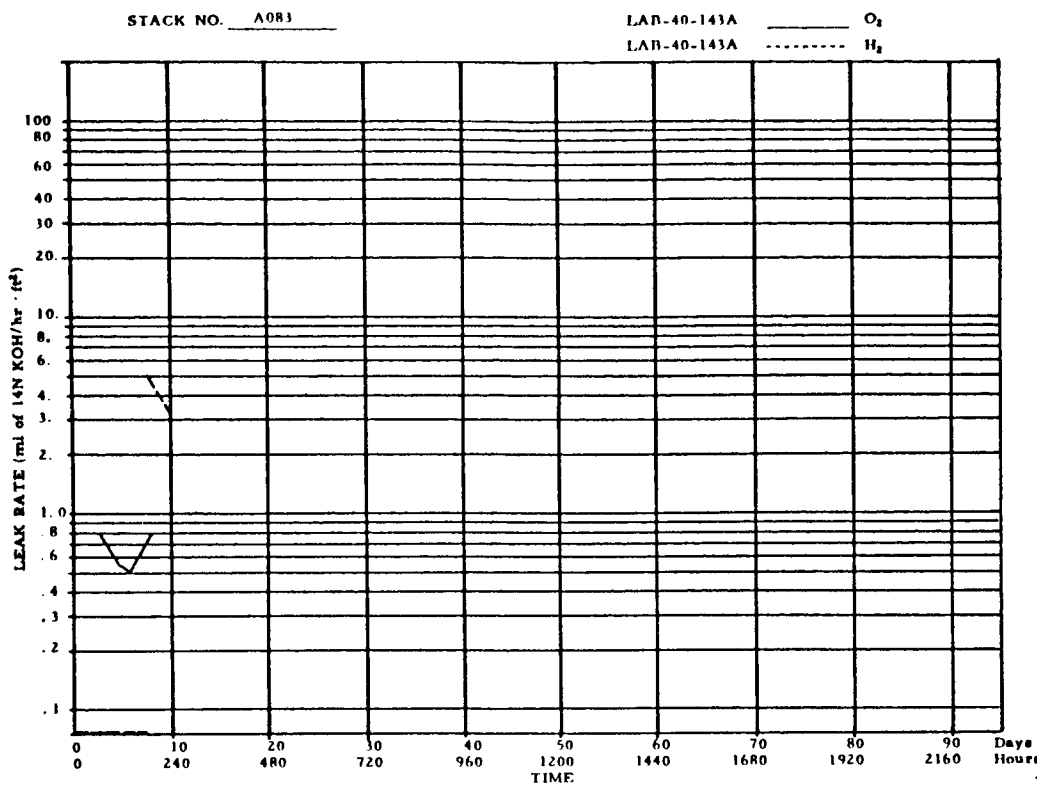
Fig. 75 - Leak Rate - Stack A-083.

D-3528
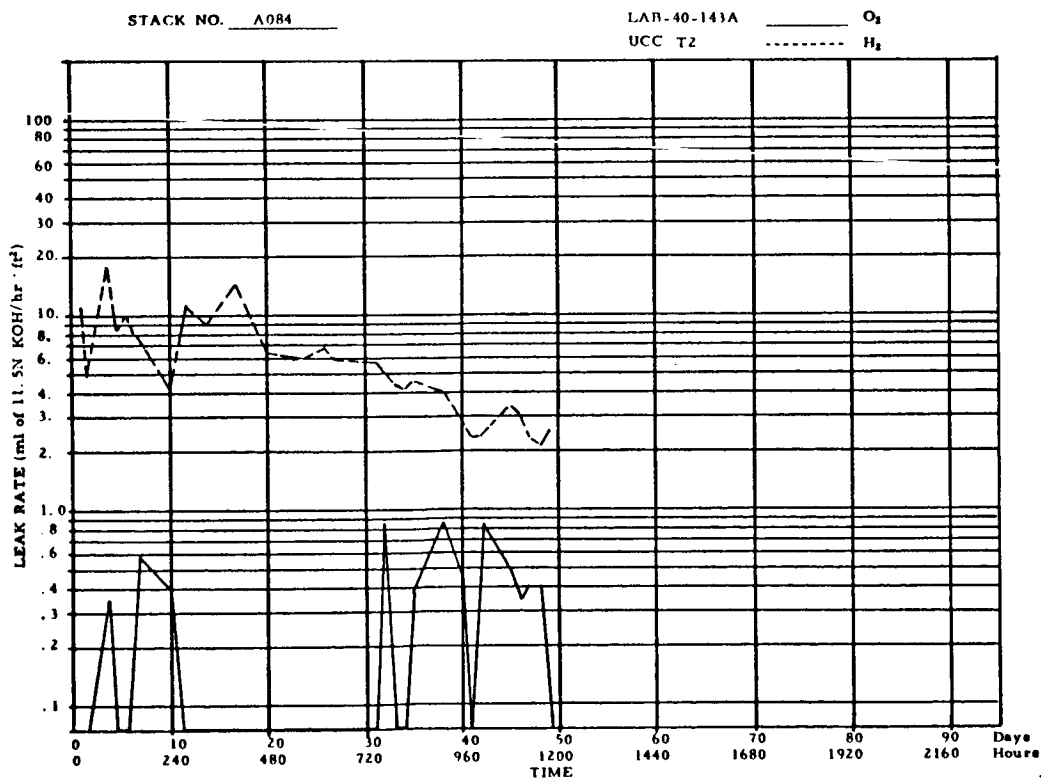
Fig. 76 - Leak Rate - Stack A-084.

D-3529
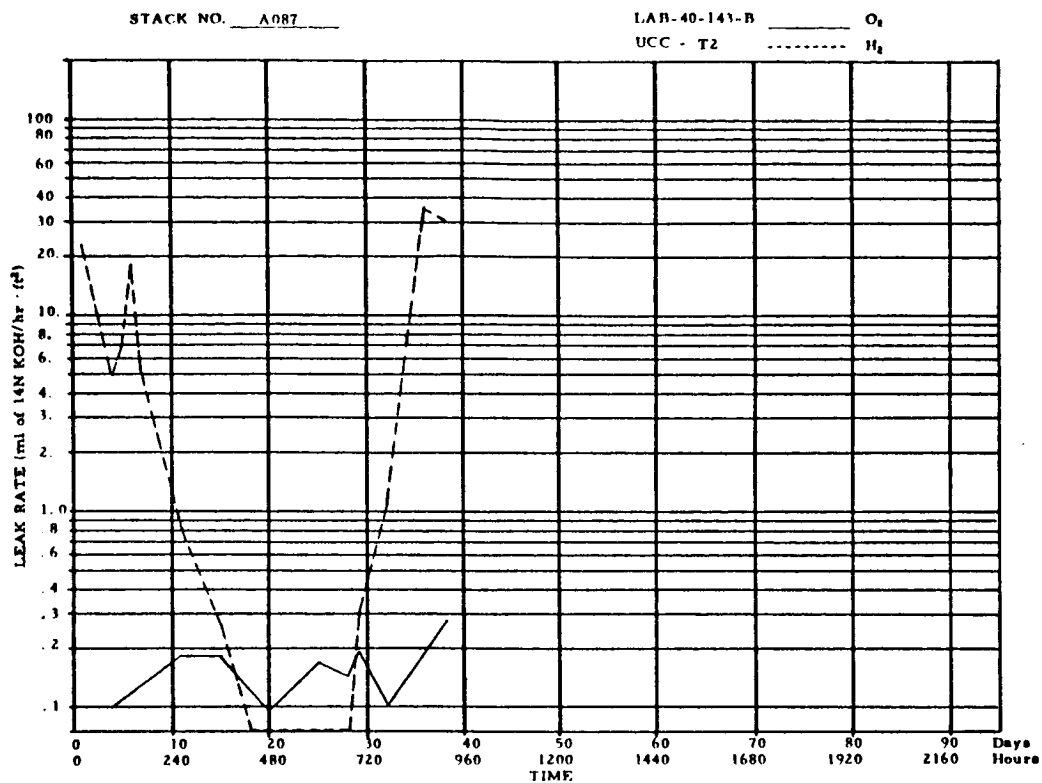
Fig. 77 - Leak Rate - Stack A-087.

D-3530

D-3531

Fig. 78 - Leak Rate - Stack A-089.

-80-
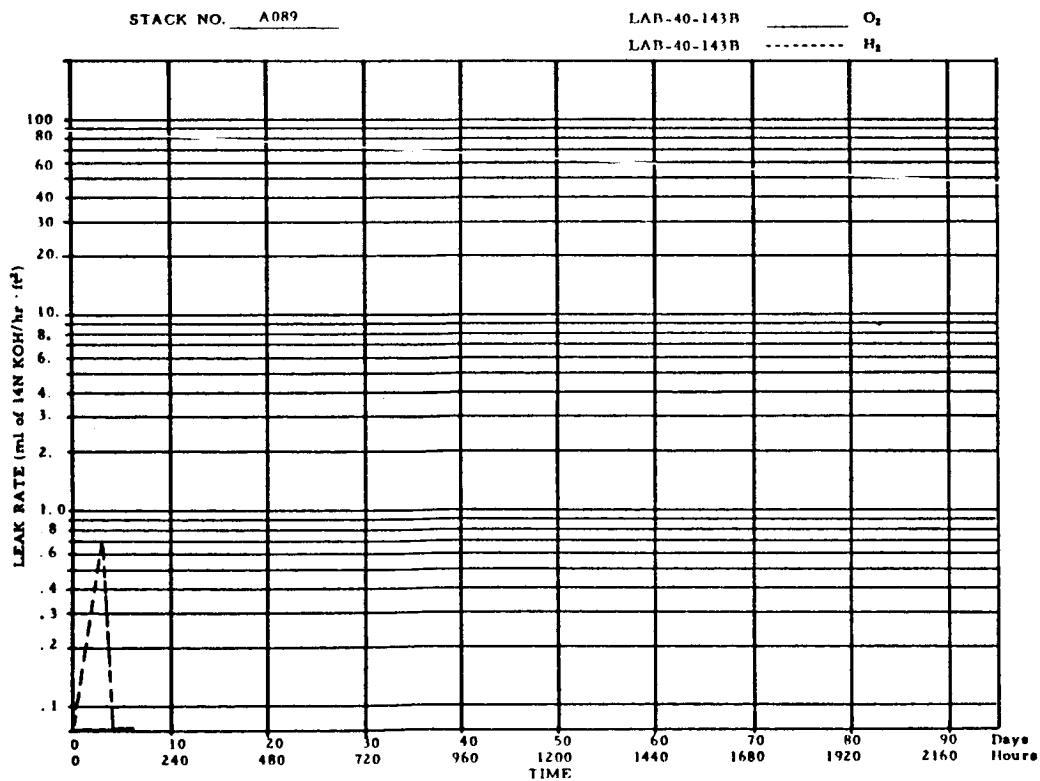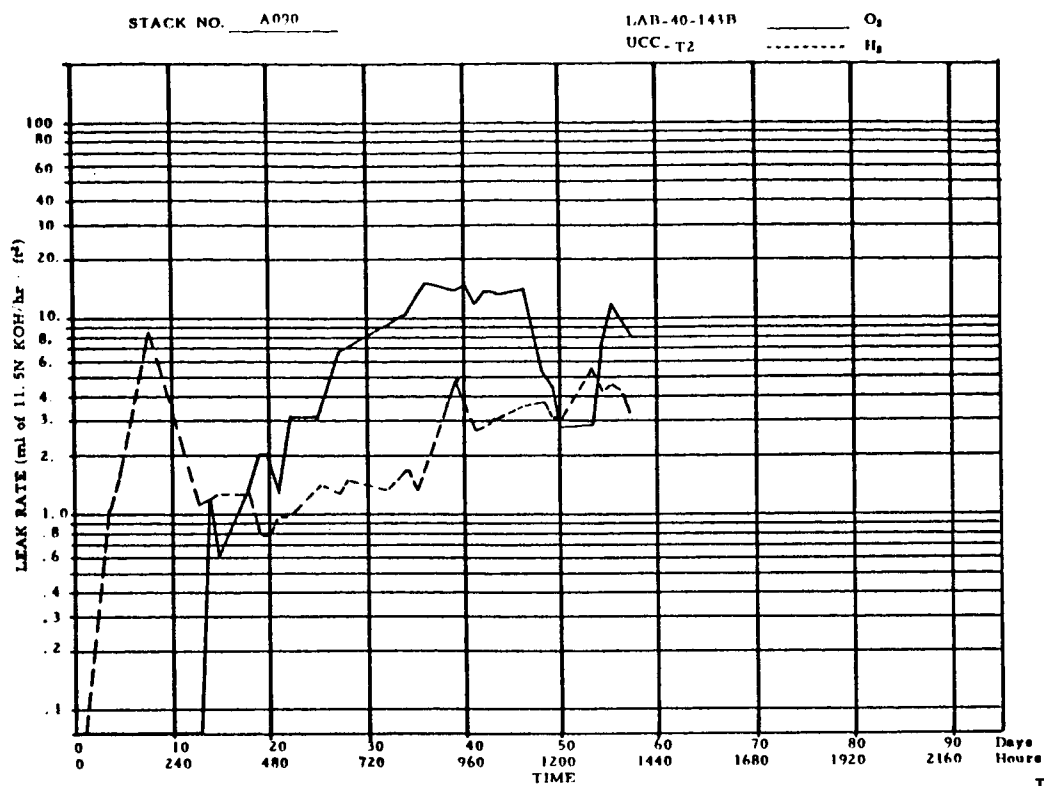
STACK NO. __A090__   LAB-40-143B _____ O$_2$
                            UCC - T2 .... H$_2$

Fig. 79 - Leak Rate - Stack A-090.

D-3532

STACK NO. __A201__   LAB-40-B-II _____ O$_2$
                            LAB-40-B-II .... H$_2$
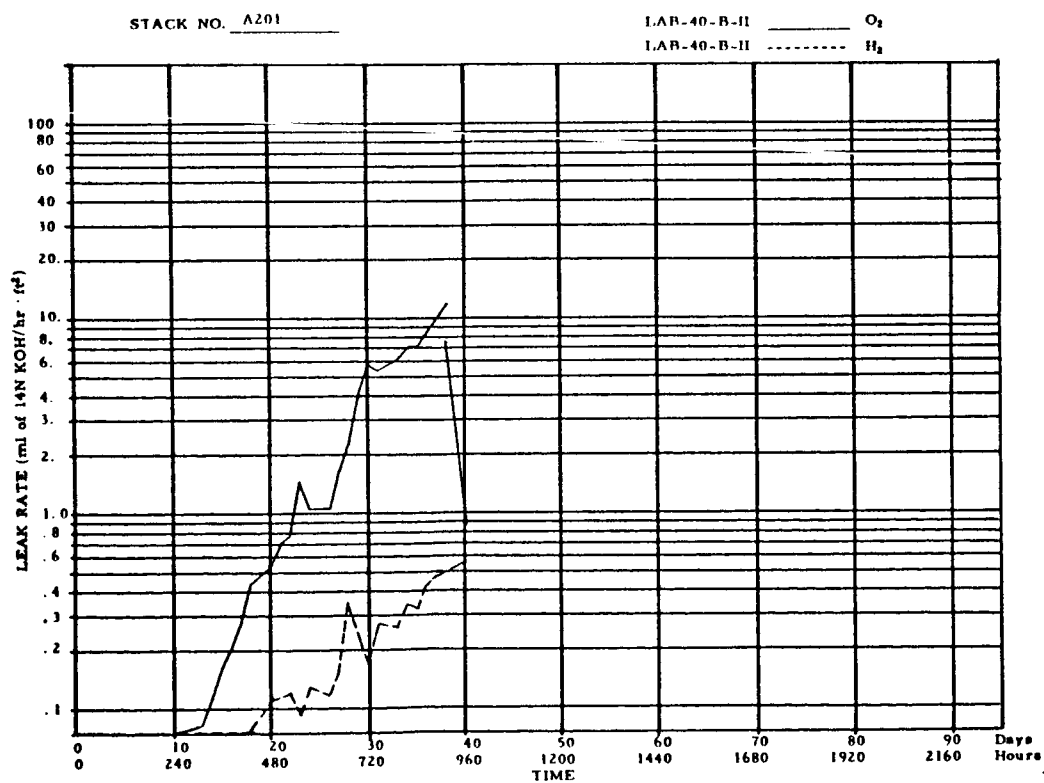
Fig. 80 - Leak Rate - Stack A-201.

D-3533

-81-
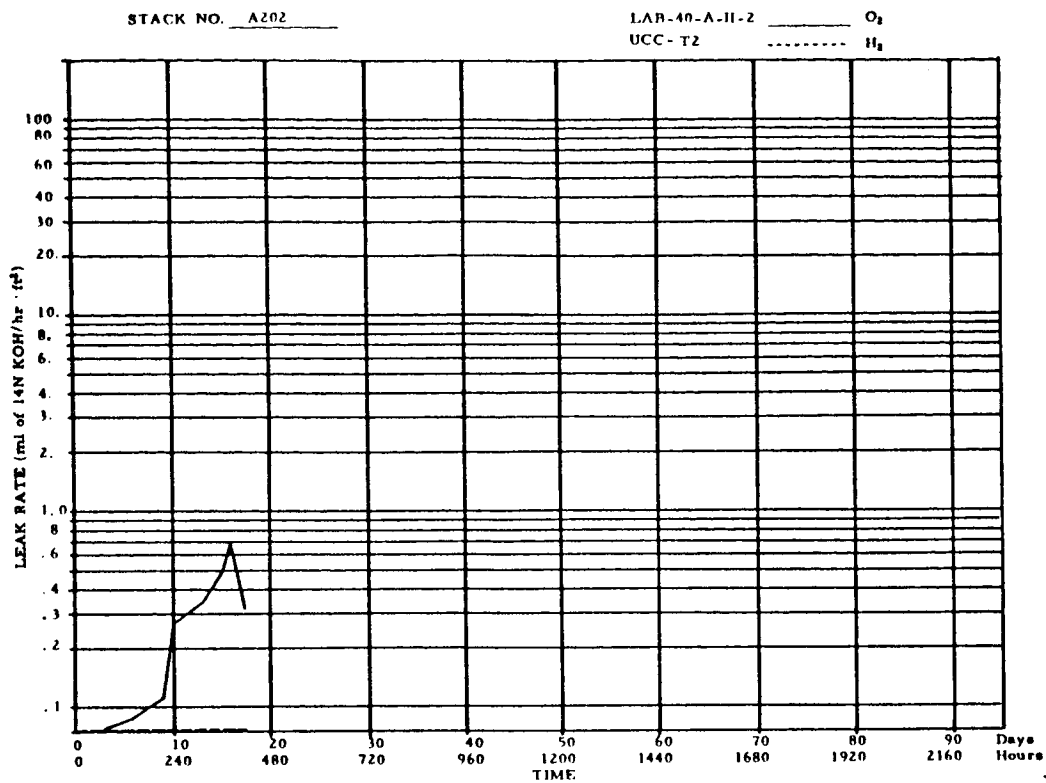
Fig. 81 - Leak Rate - Stack A-202.

D-3534
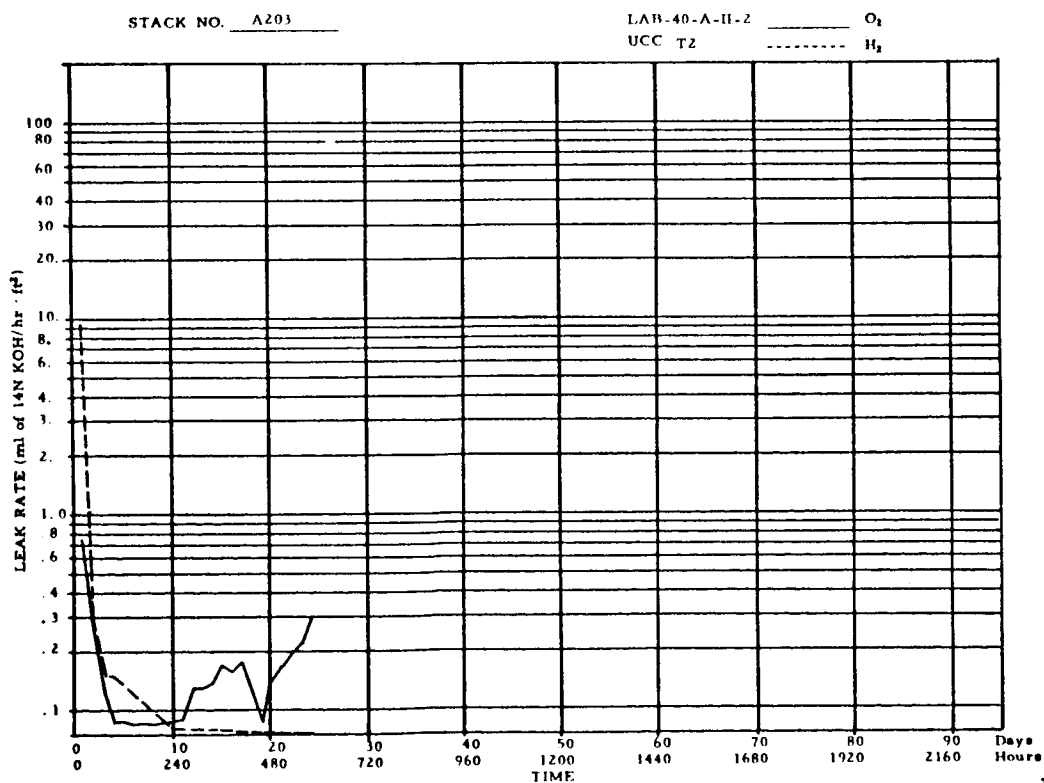
Fig. 82 - Leak Rate - Stack A-203.

D-3535
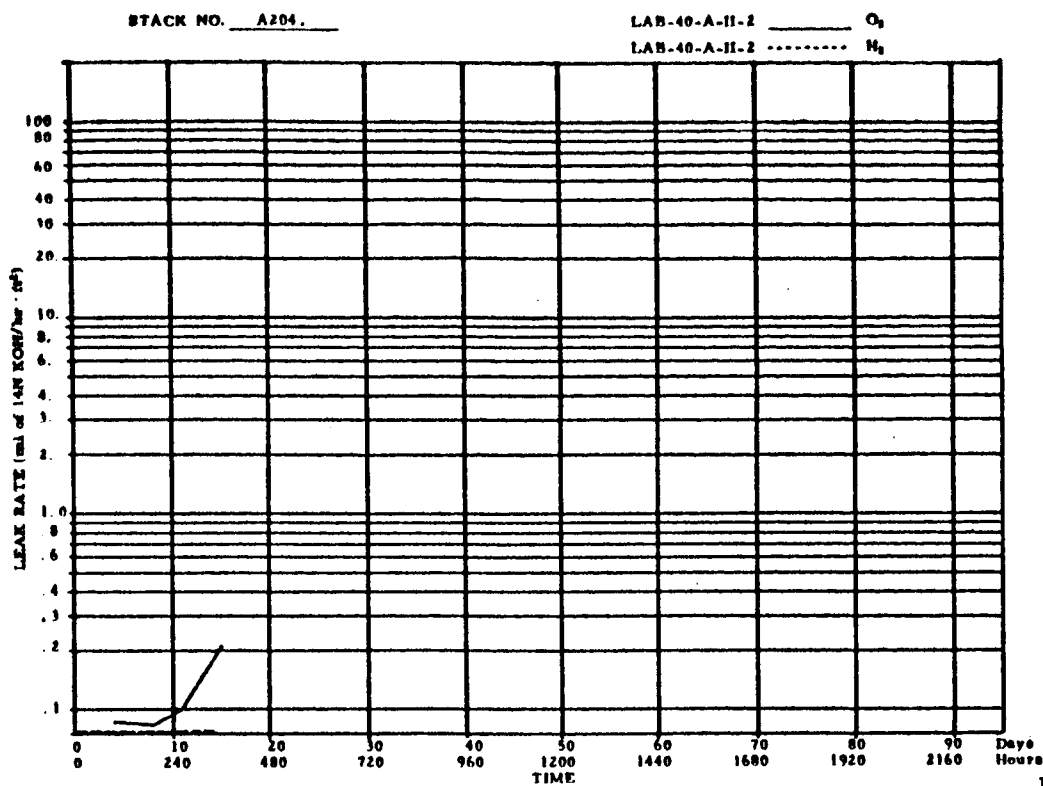
D-3536

Fig. 83 - Leak Rate - Stack A-204.
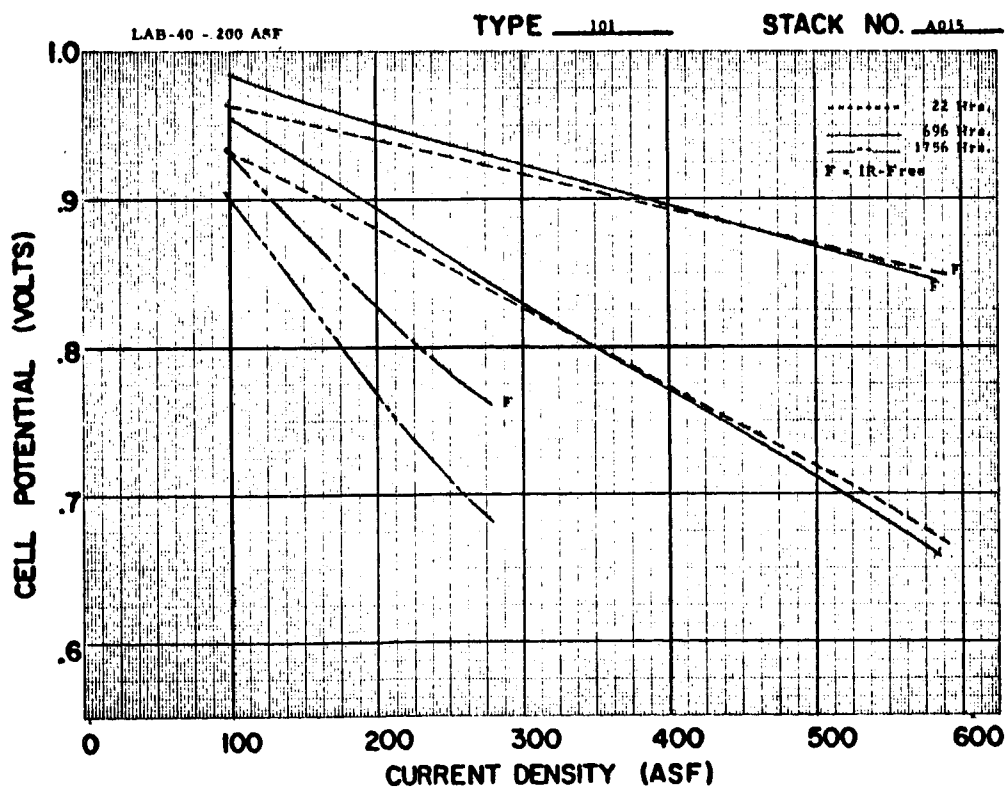
D-3537

Fig. 84 - Polarization Curves - Stack A-015, Normal.
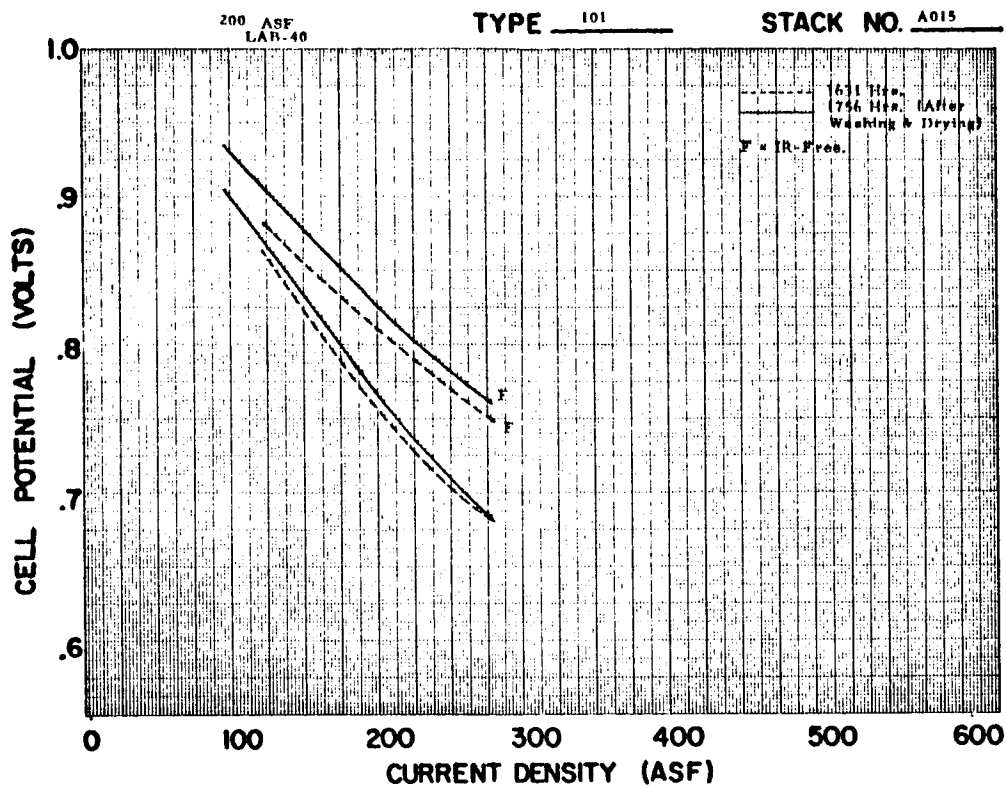
D-3538

Fig. 85 - Polarization Curves - Stack A-015,
Before and After Washing and Drying.
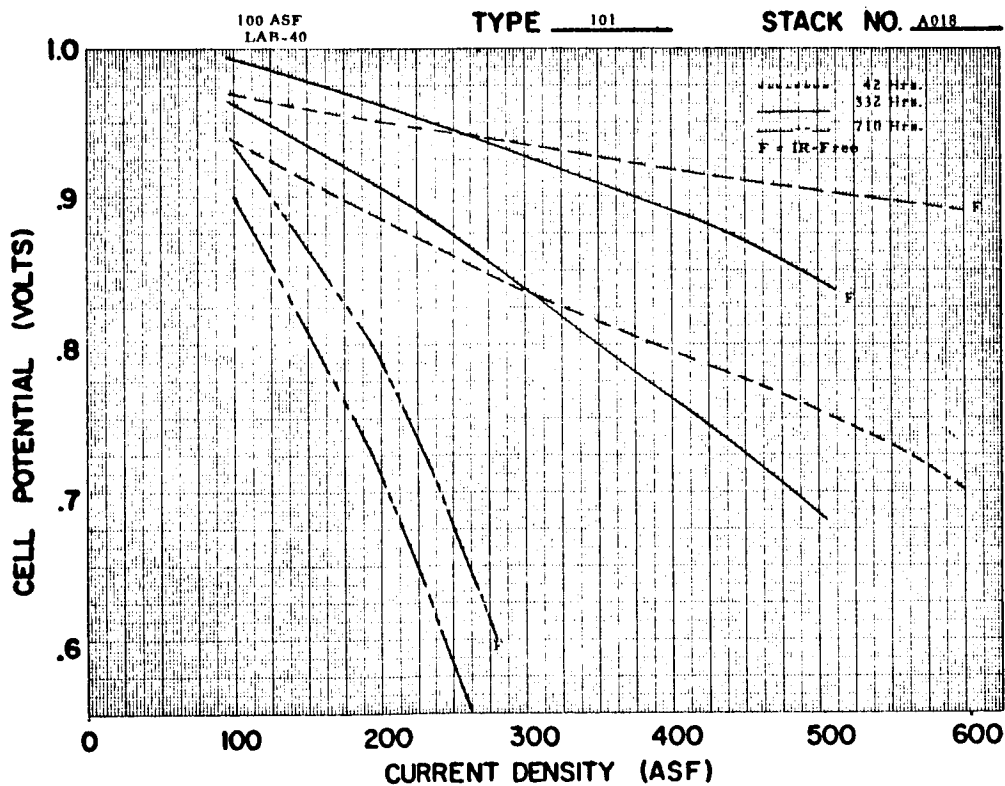
D-3539

Fig. 86 - Polarization Curves - Stack A-018.

-84-
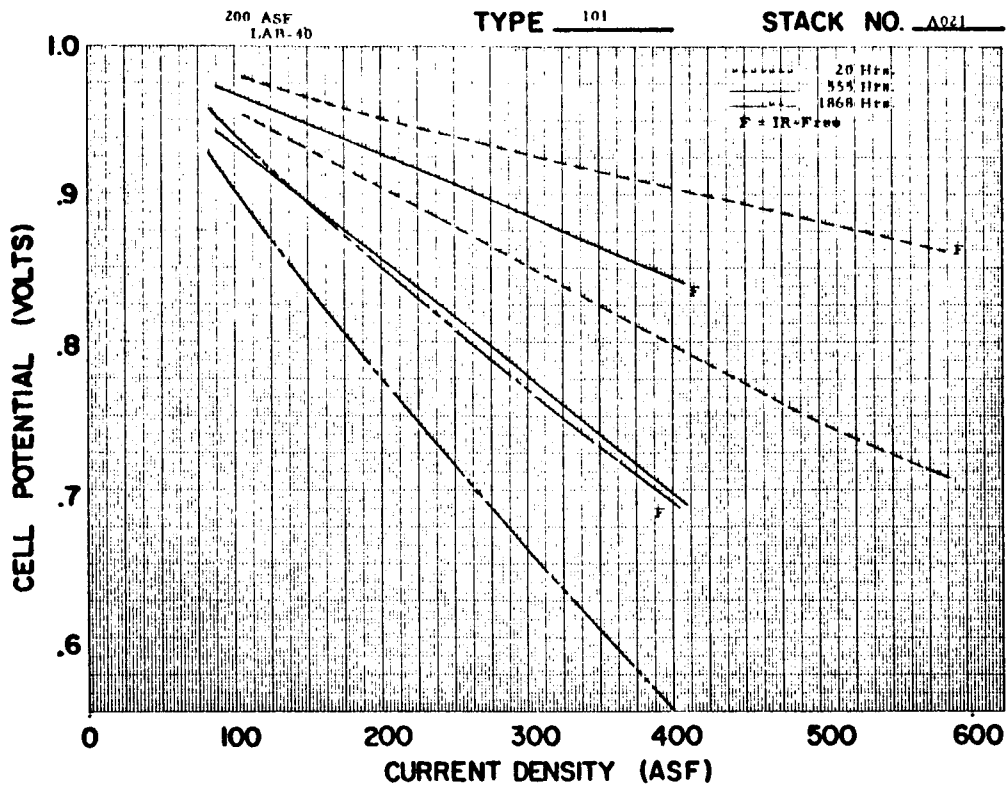
Fig. 87 - Polarization Curves - Stack A-021.

D-3540

Fig. 88 - Polarization Curves - Stack A-022, Normal.

D-3541

200 ASF
LAB-40

199 Hrs.
296 Hrs. (After Washing &
Drying)
F - IR-Free
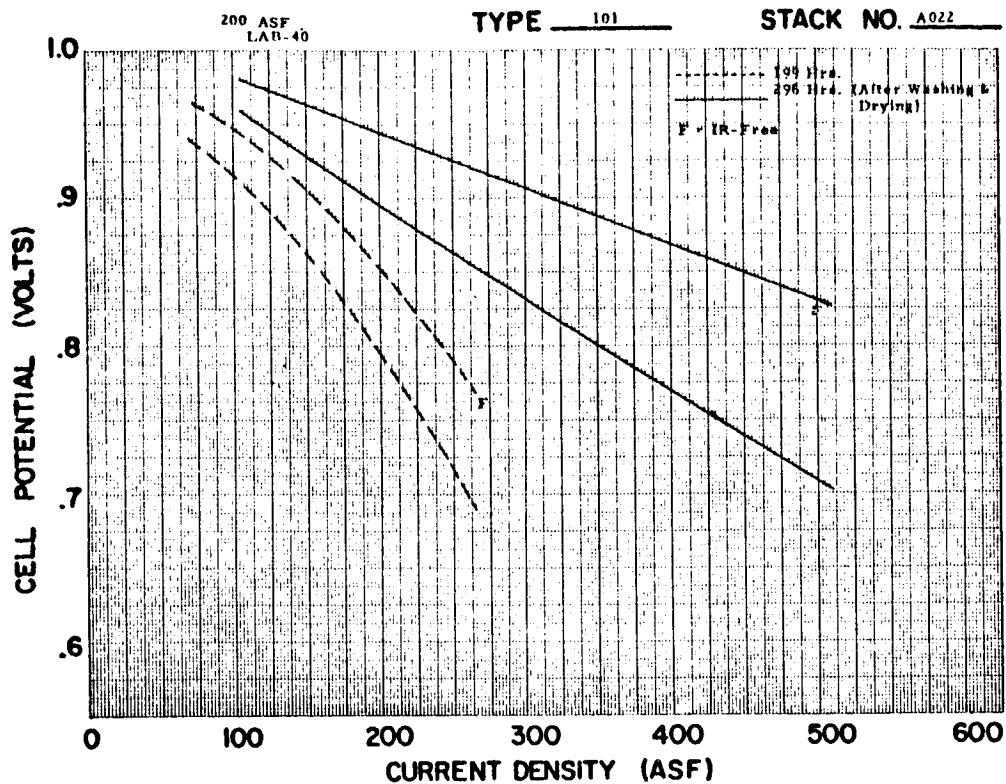
D-3542

Fig. 89 - Polarization Curves - Stack A-022,
Before and After Washing and Drying.

100 ASF
LAB-40/T2 Hybrid

5 Hrs.
459 Hrs.
857 Hrs.
F - IR-Free
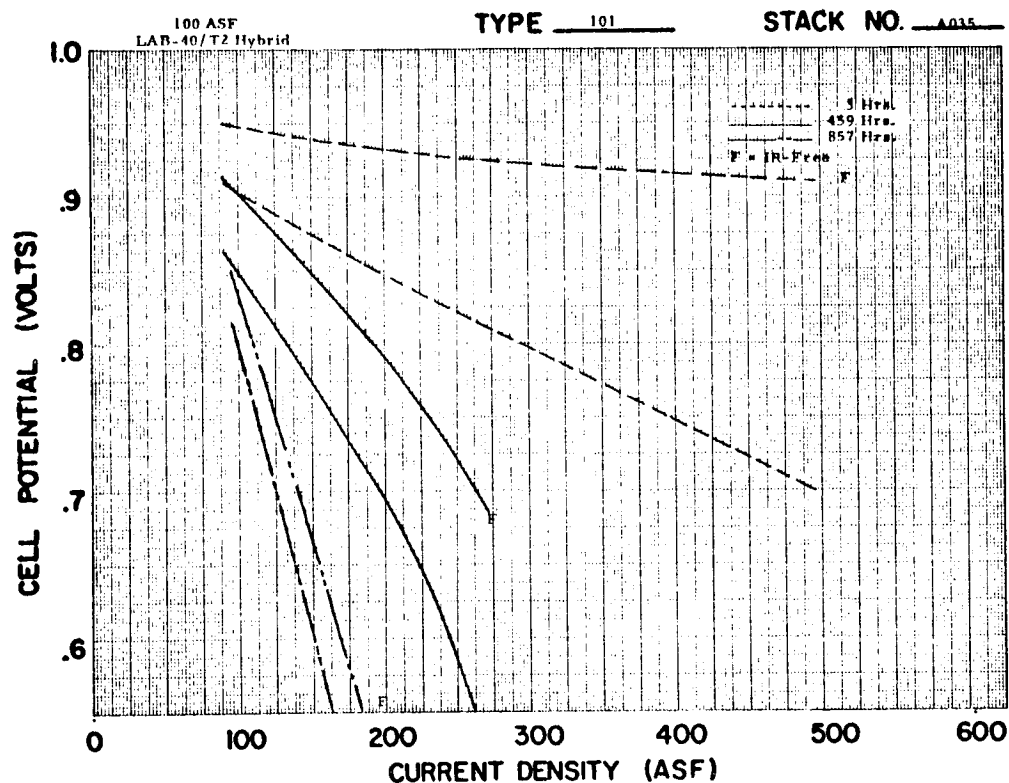
D-3543

Fig. 90 - Polarization Curves - Stack A-035, Normal.
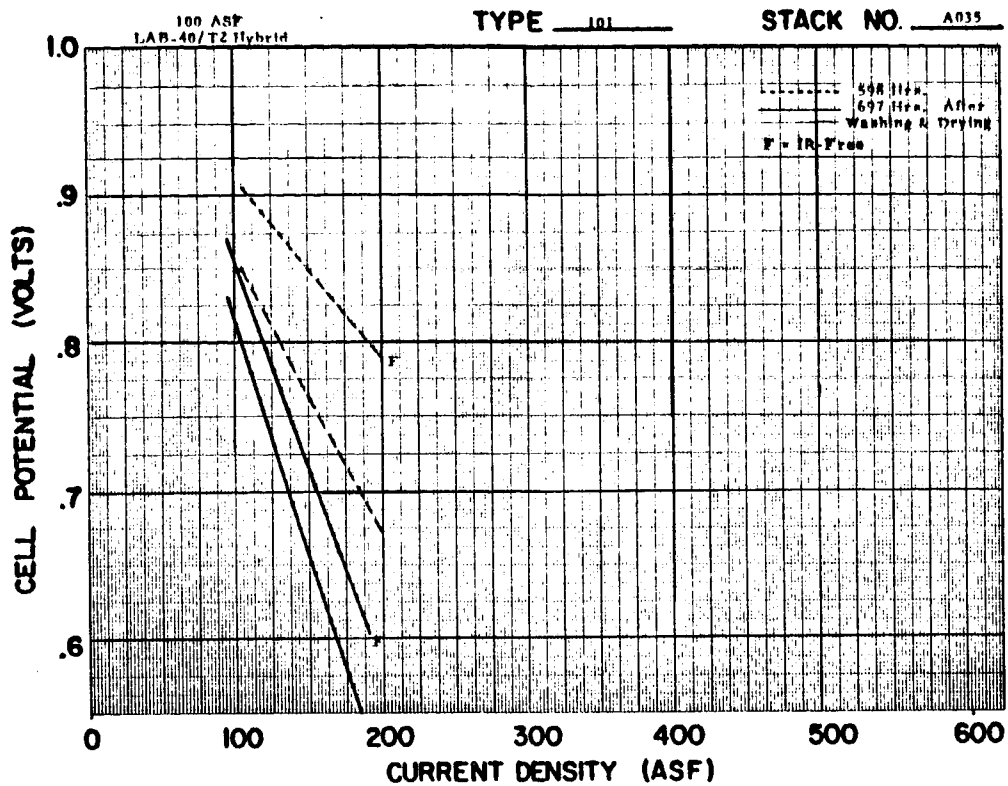
Fig. 91 - Polarization Curves - Stack A-035,
Before and After Washing and Drying.

D-3544
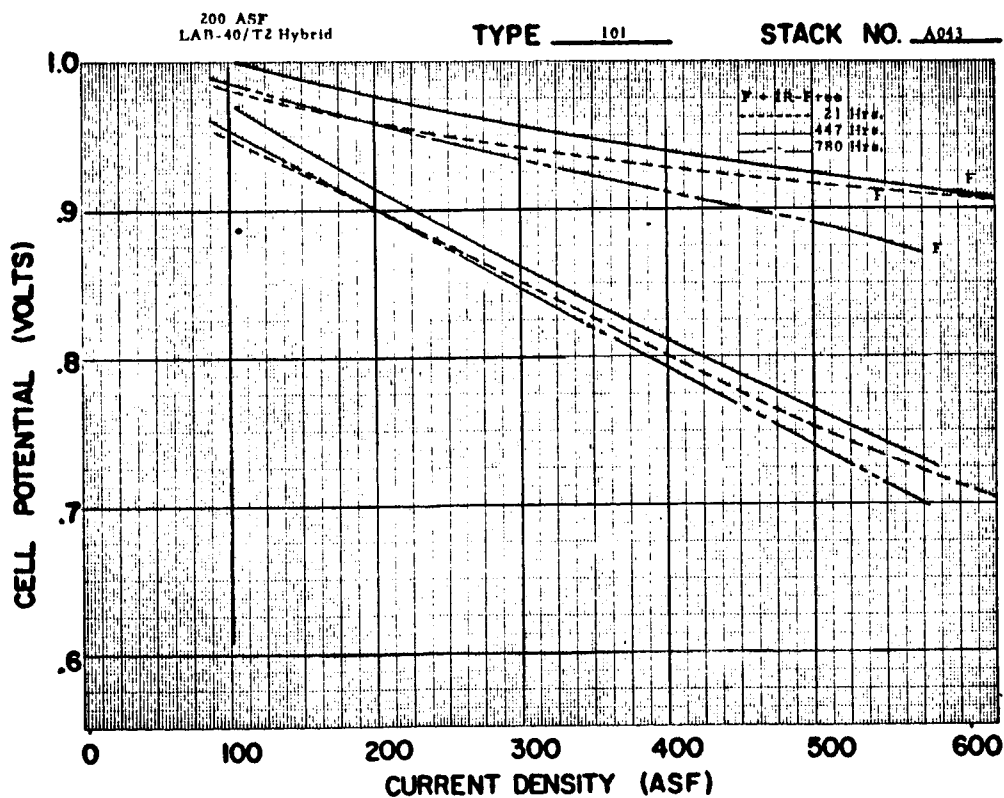
Fig. 92 - Polarization Curves - Stack A-043.

D-3545
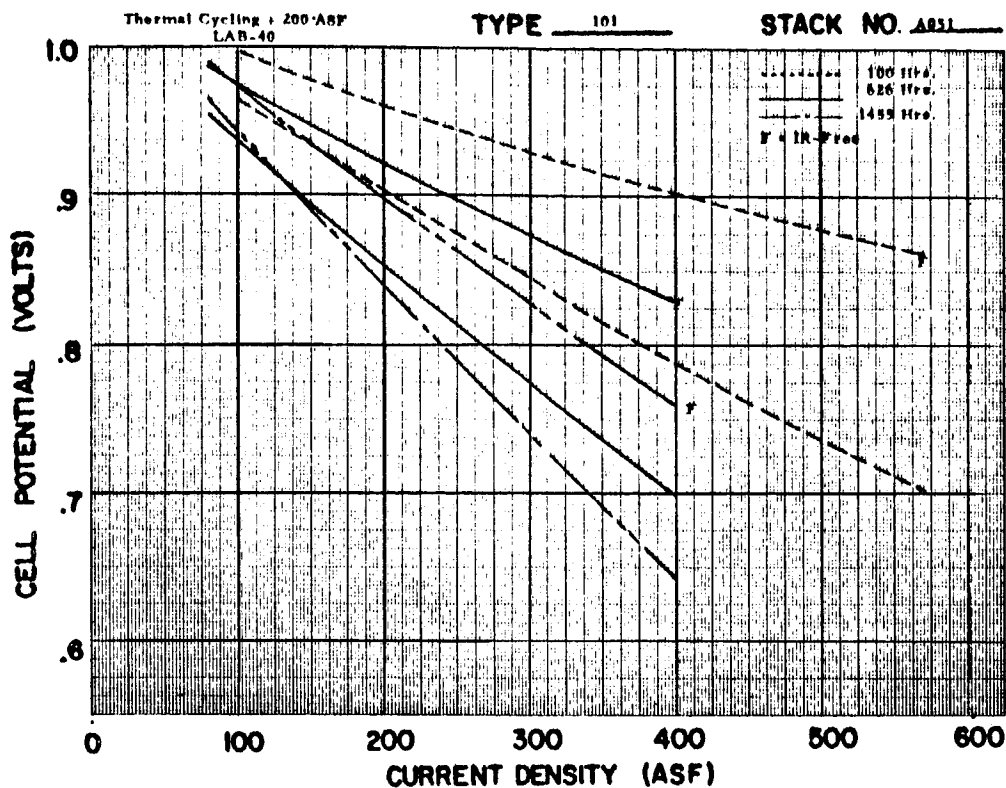
Fig. 93 - Polarization Curves - Stack A-051.

D-3546
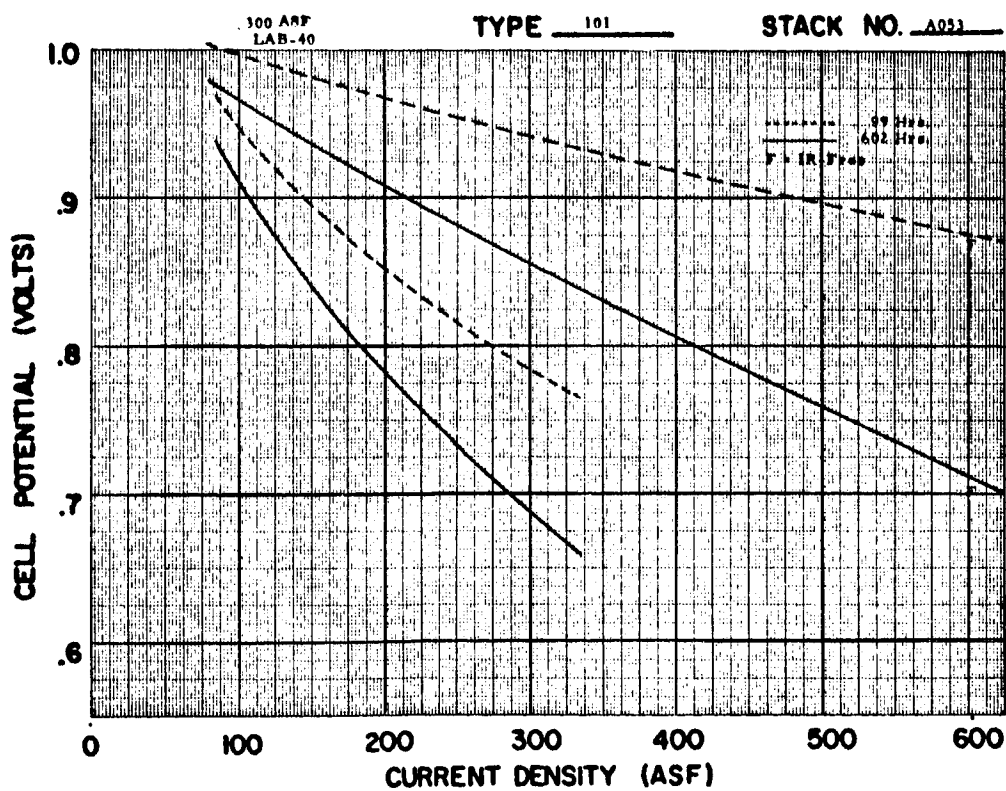
Fig. 94 - Polarization Curves - Stack A-053.

D-3547
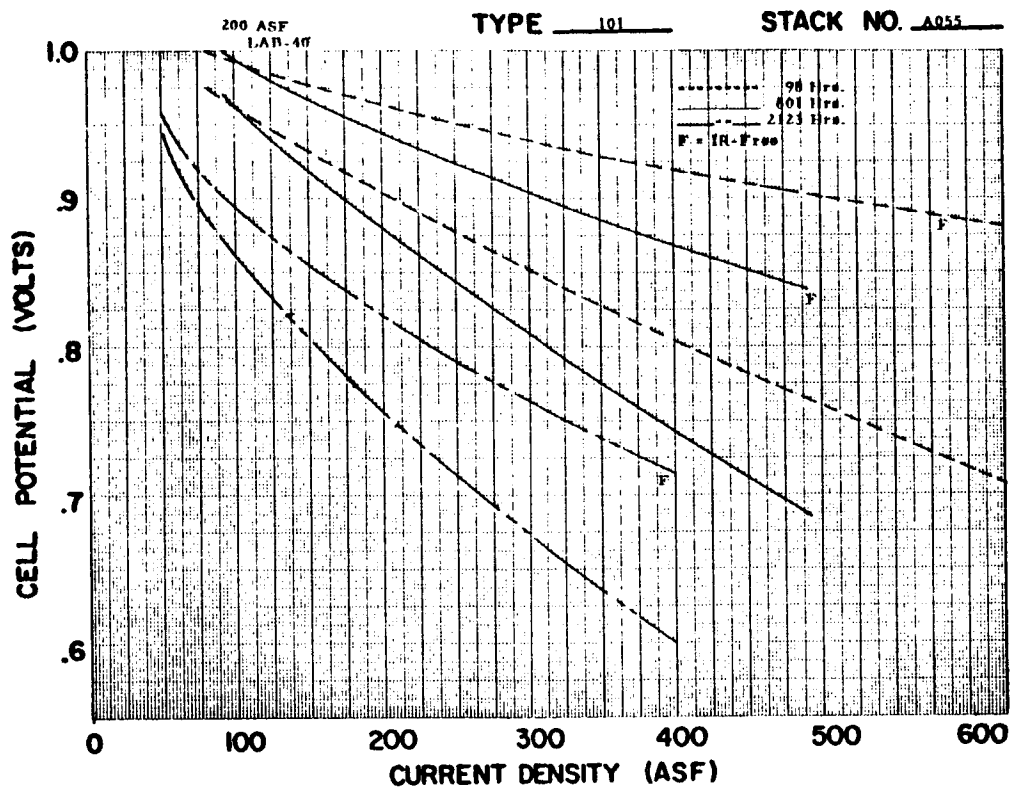
-88-

Fig. 95 - Polarization Curves - Stack A-055.

D-3548
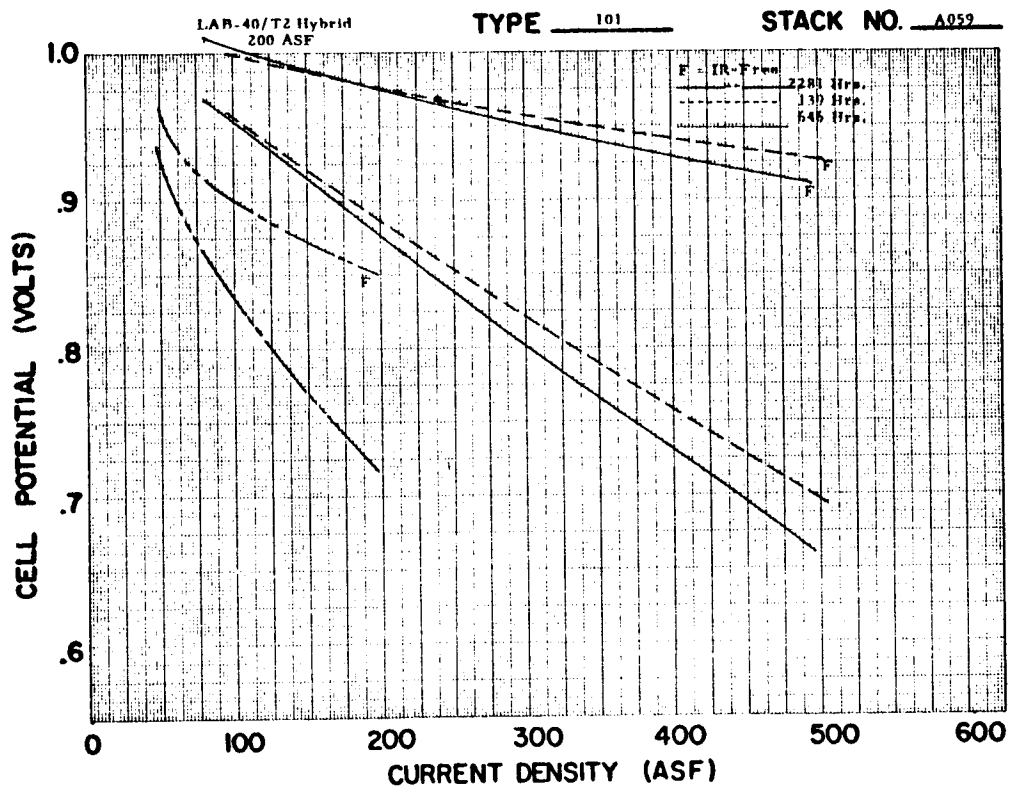
Fig. 96 - Polarization Curves - Stack A-059.

D-3549

-89-
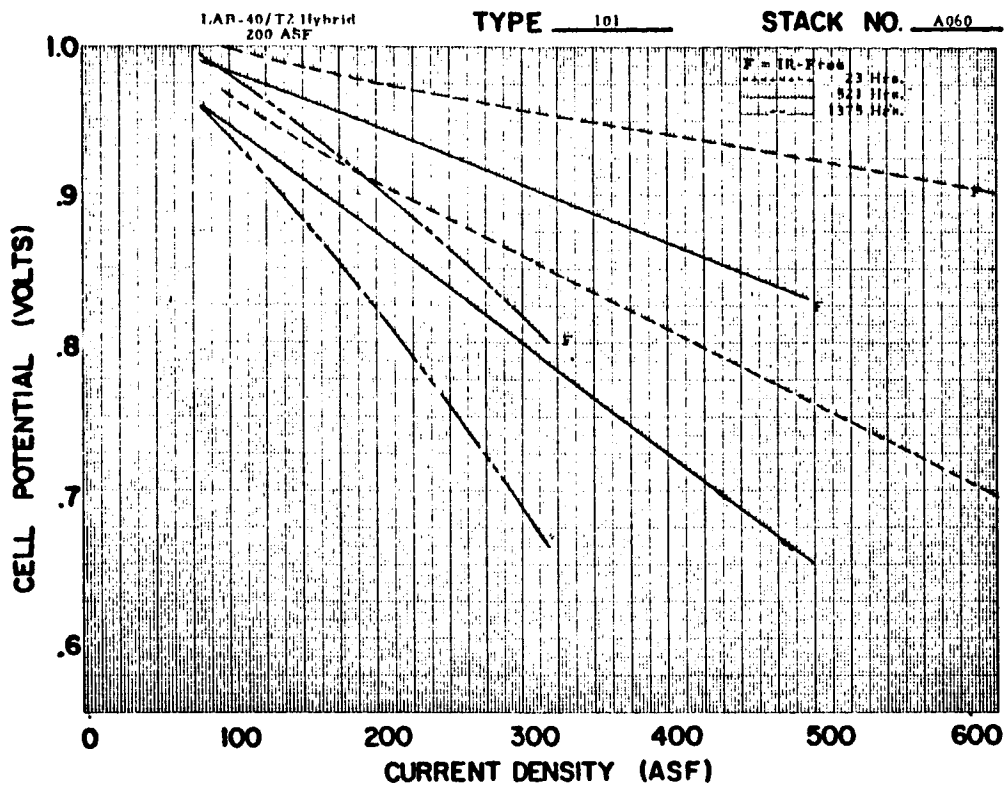
Fig. 97 - Polarization Curves - Stack A-060.
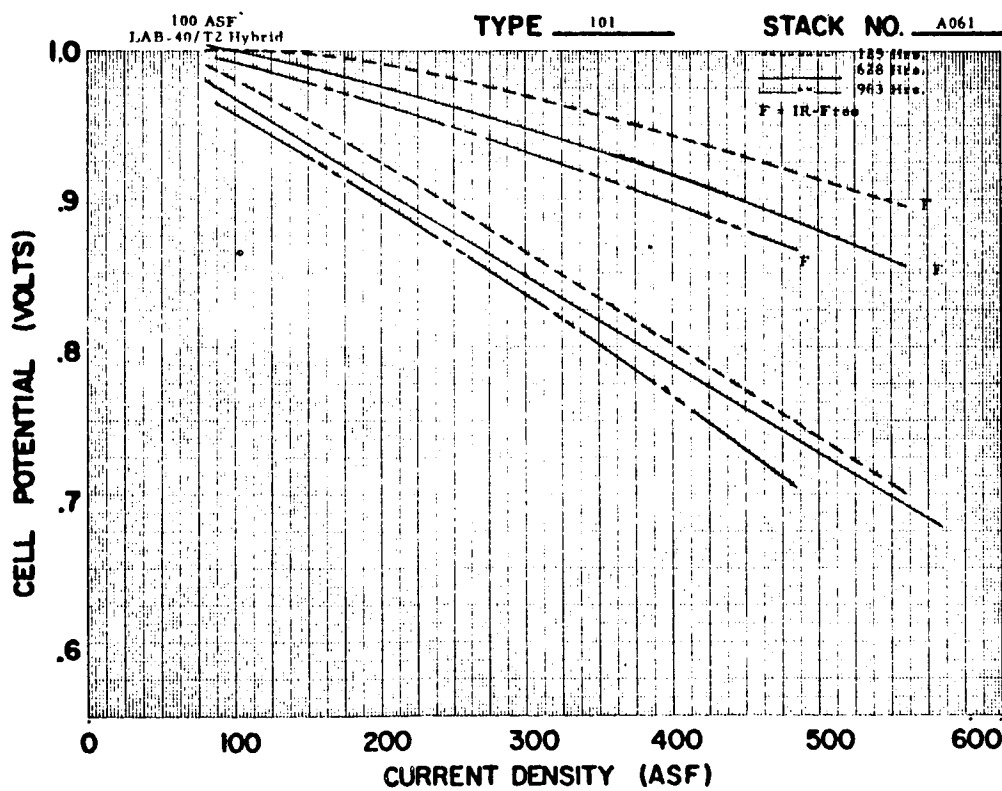
Fig. 98 - Polarization Curves - Stack A-061.

-90-

CELL POTENTIAL (VOLTS)

CURRENT DENSITY (ASF)

D-3552

Fig. 99 - Polarization Curves - Stack A-066.

CELL POTENTIAL (VOLTS)

CURRENT DENSITY (ASF)

D-3553
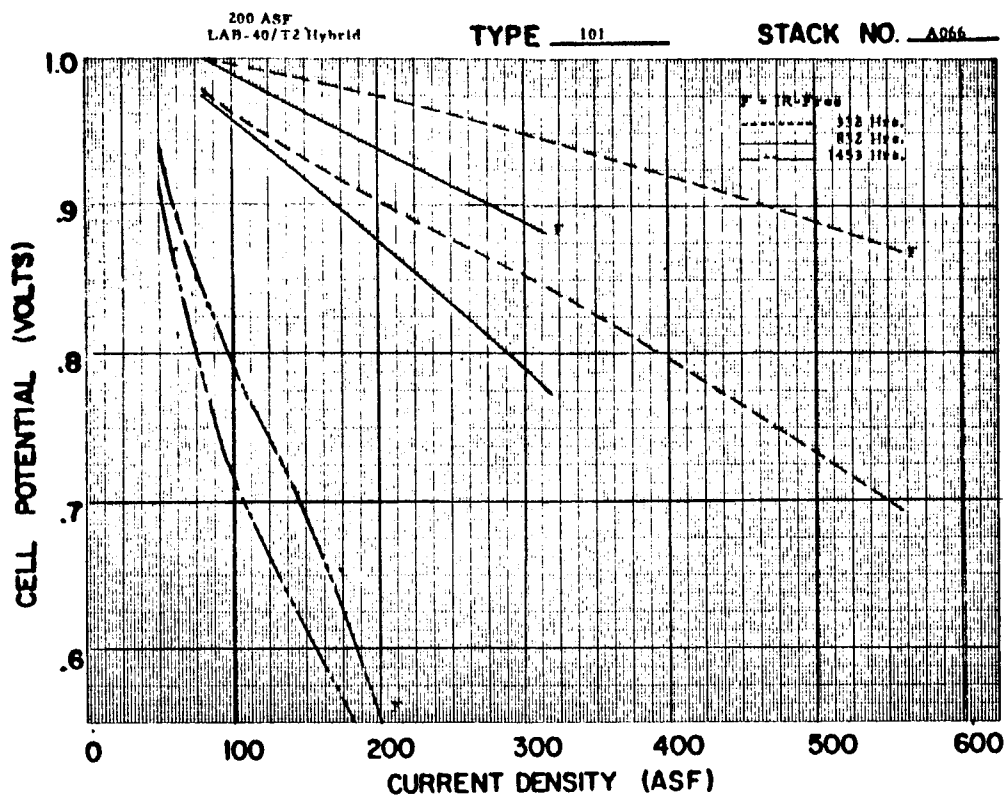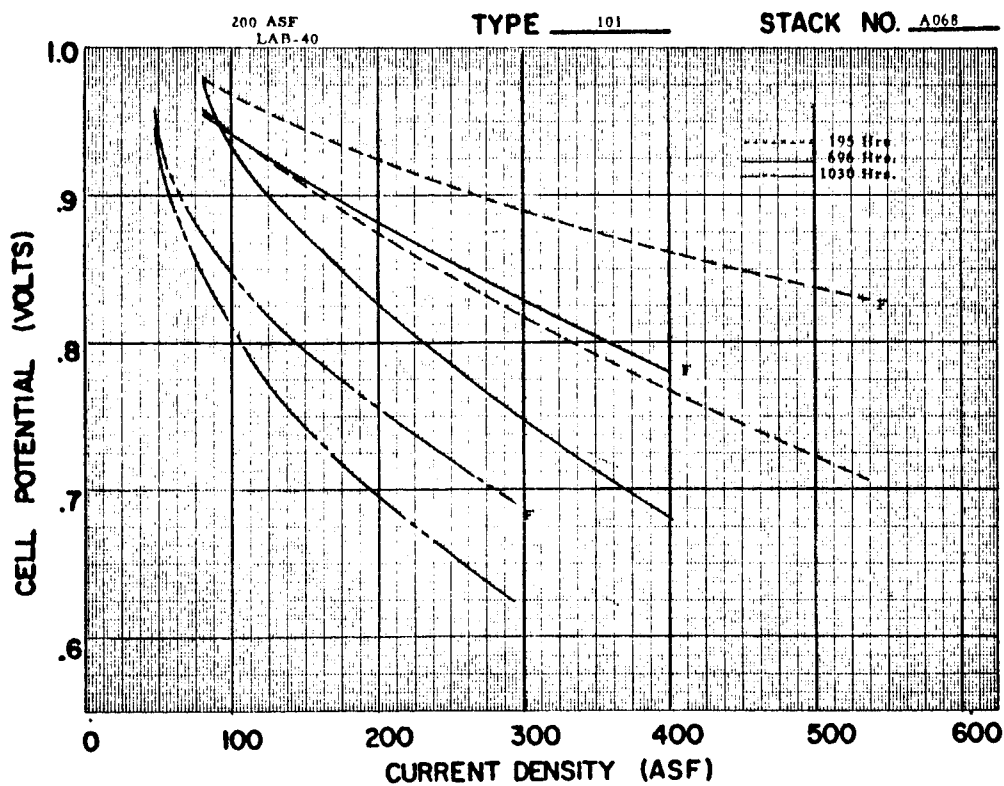
Fig. 100 - Polarization Curves - Stack A-068.
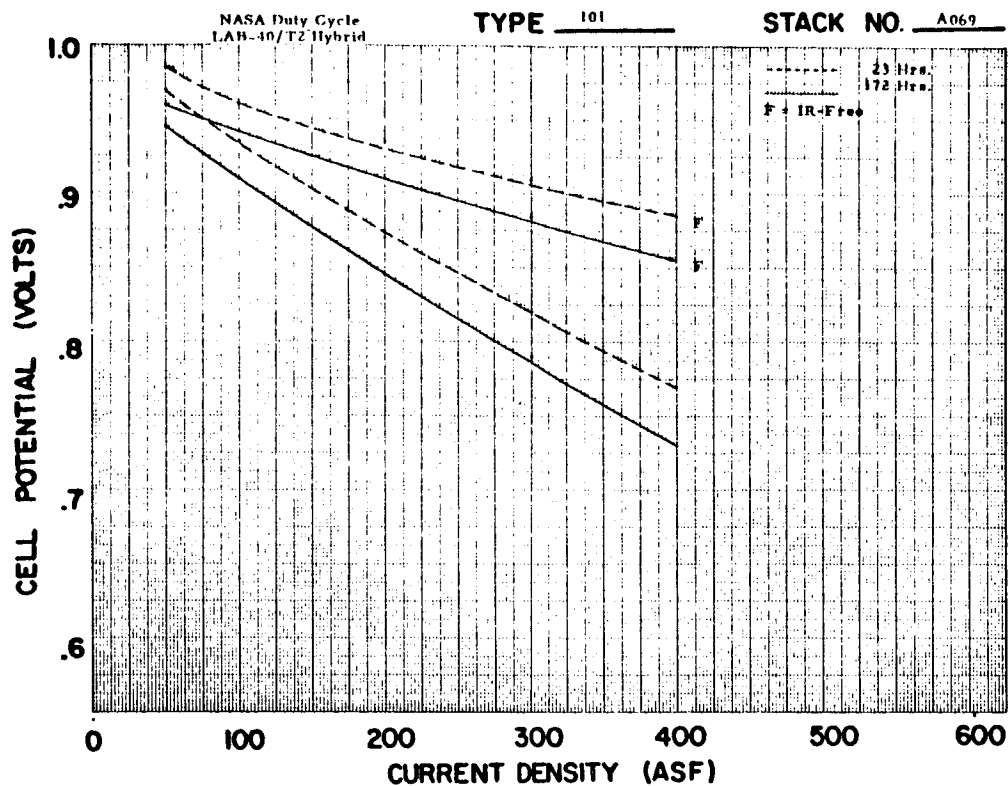
Fig. 101 - Polarization Curves - Stack A-069.

D-3554
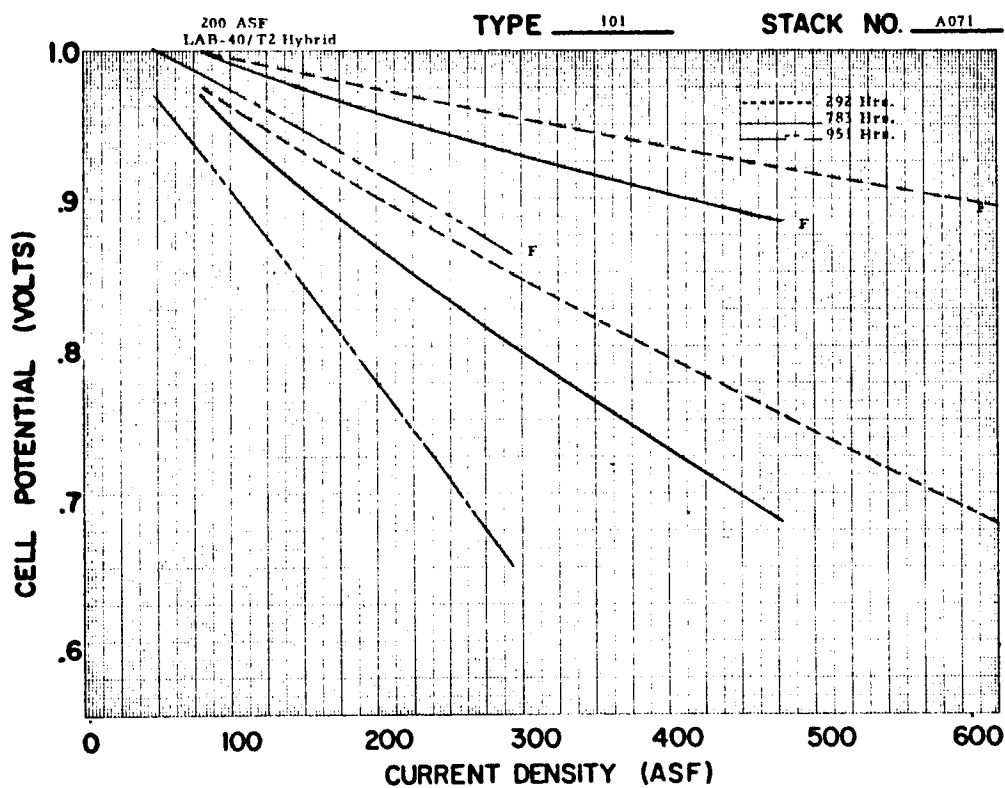
Fig. 102 - Polarization Curves - Stack A-071.

D-3555

100 ASF
LAB-40/T2 Hybrid

------- 123 Hrs.
F = IR-Free

F

CELL POTENTIAL (VOLTS)

1.0
.9
.8
.7
.6

0   100   200   300   400   500   600
CURRENT DENSITY (ASF)
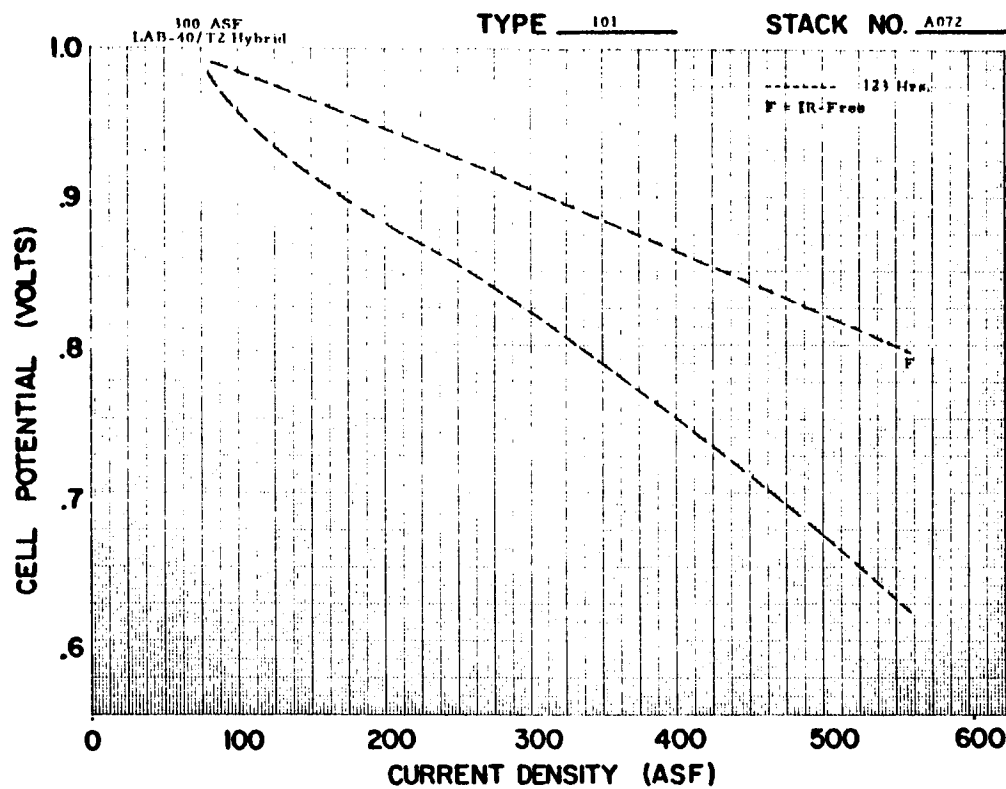
D-3556

Fig. 103 - Polarization Curves - Stack A-072.

NASA Duty Cycle
LAB-40/T2 Hybrid

------- 18 Hrs.
——— 607 Hrs.
F = IR-Free

F

F

CELL POTENTIAL (VOLTS)

1.0
.9
.8
.7
.6

0   100   200   300   400   500   600
CURRENT DENSITY (ASF)
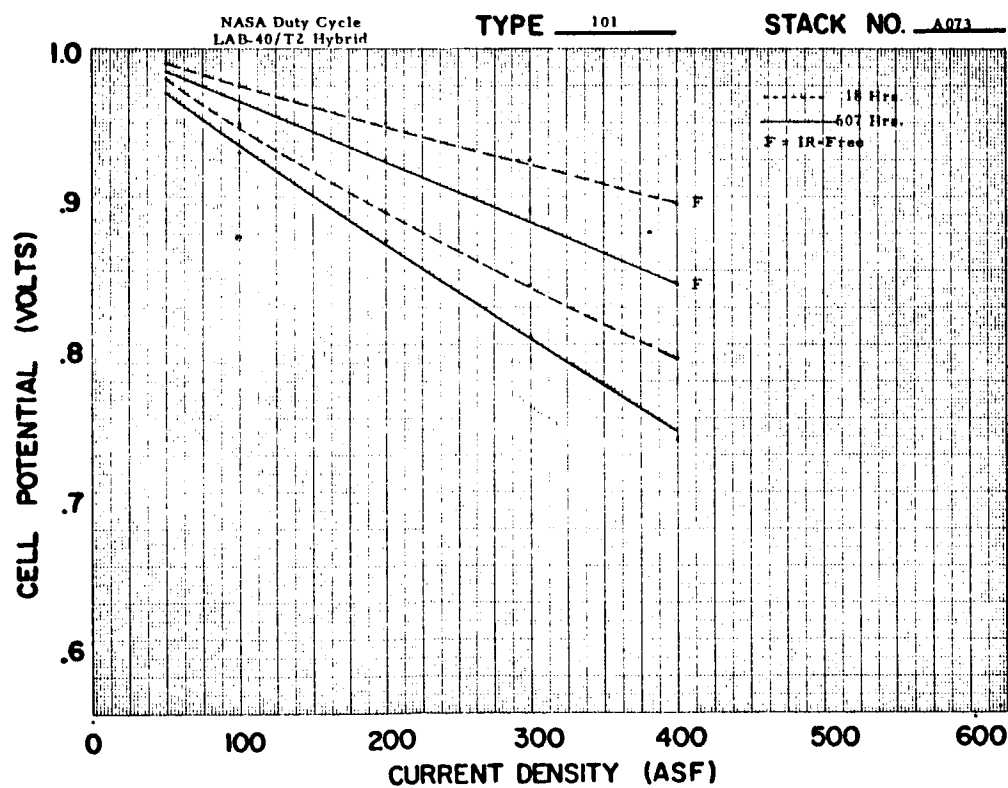
D-3557

Fig. 104 - Polarization Curves - Stack A-073.

-93-
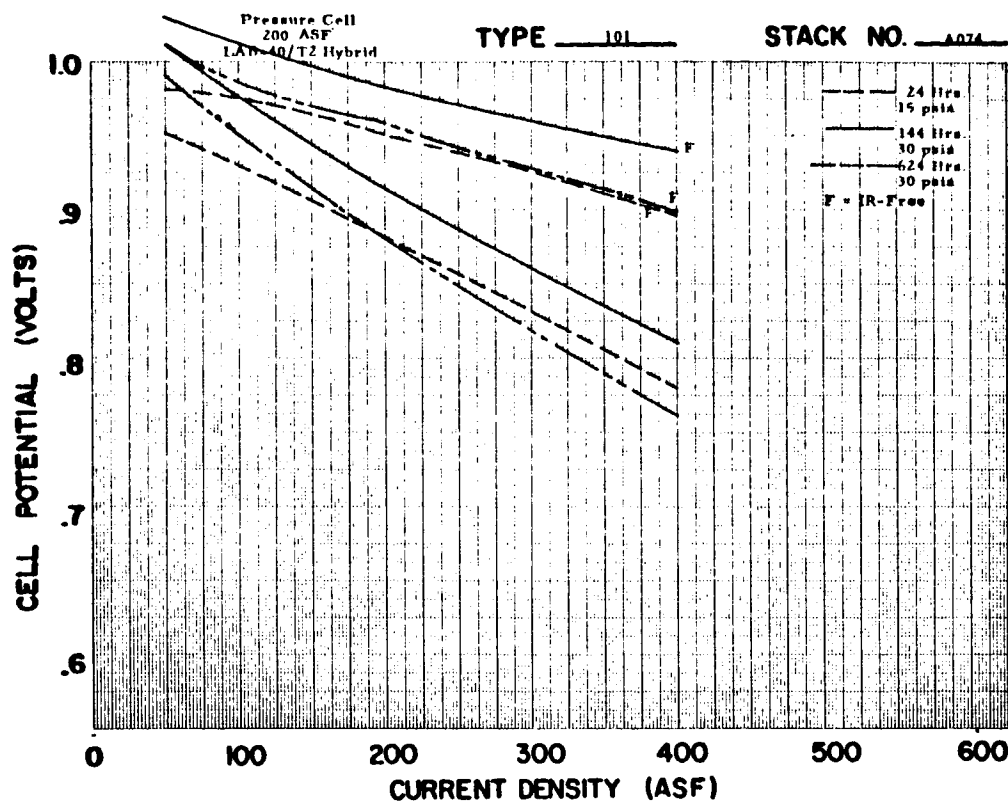
Fig. 105 - Polarization Curves - Stack A-074, Normal, at 30 psia.
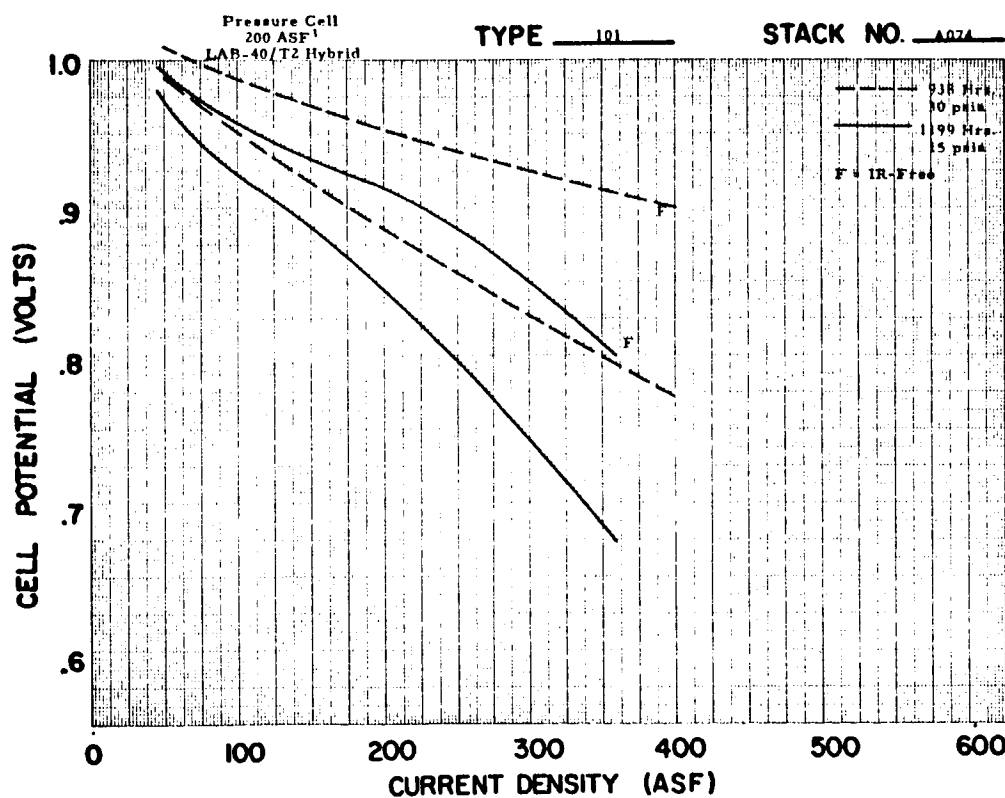
Fig. 106 - Polarization Curves - Stack A-074, at 30 and 15 psia.
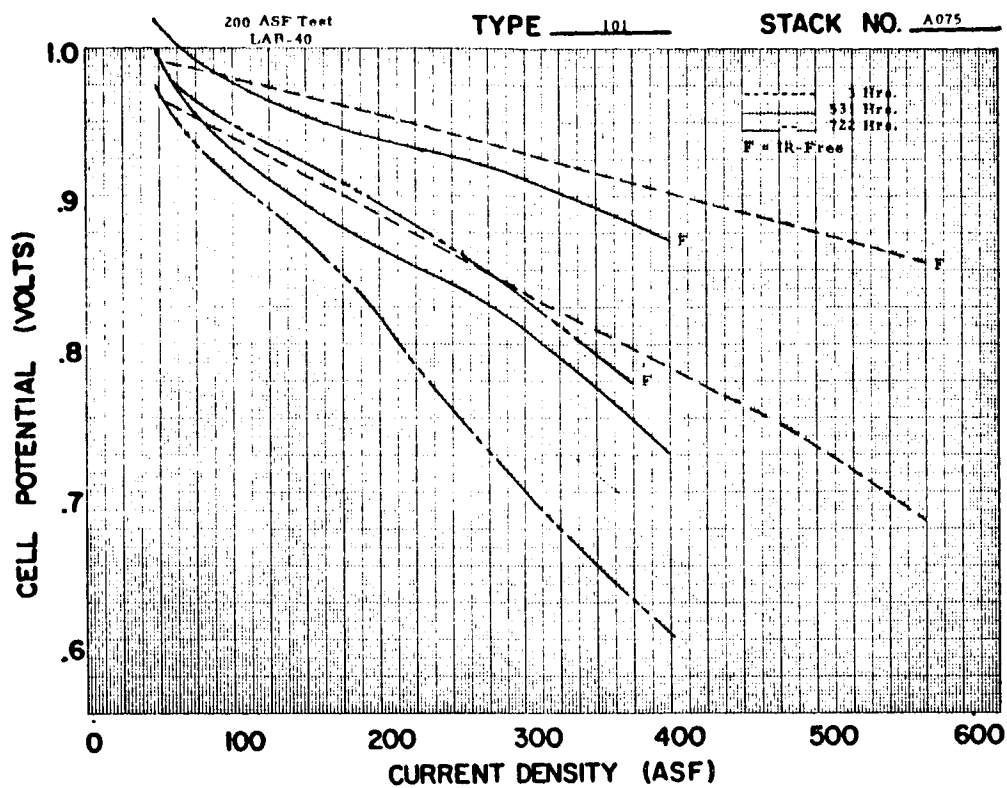
Fig. 107 - Polarization Curves - Stack A-075.
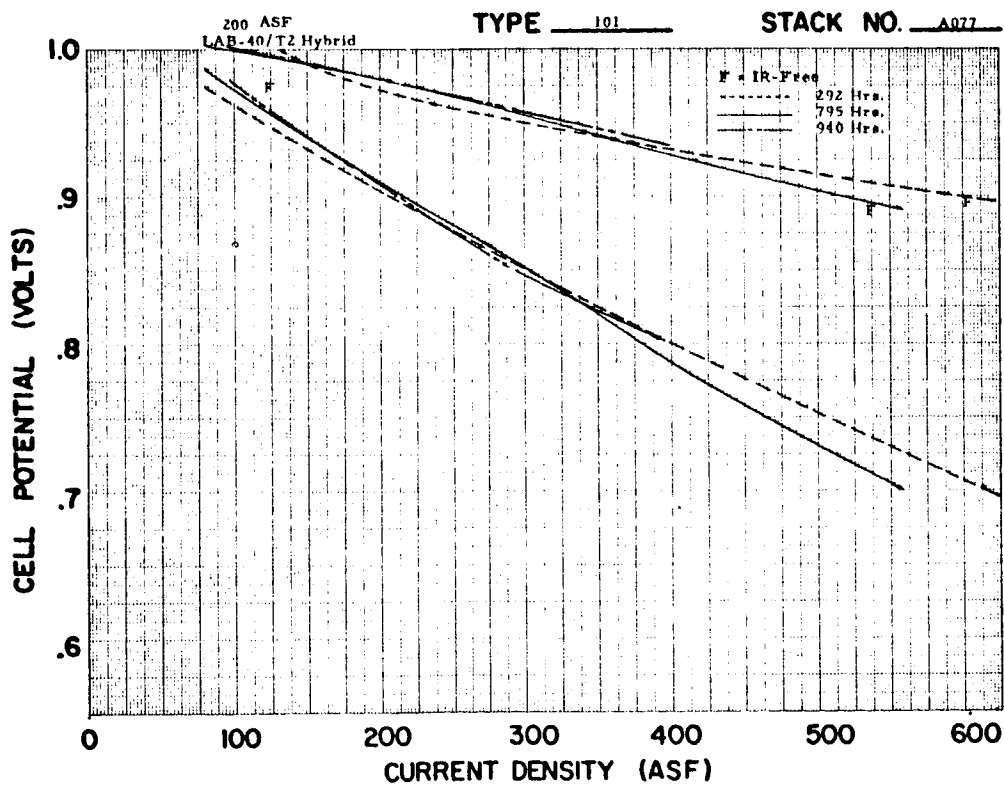
Fig. 108 - Polarization Curves - Stack A-077.

Pressure Cell
200 ASF
LAB-40/T2 Hybrid

TYPE 101
STACK NO. 078

99 Hrs.
30 psia

650 Hrs.
30 psia

1655 Hrs.
30 psia

F = IR-Free

CURRENT DENSITY (ASF)
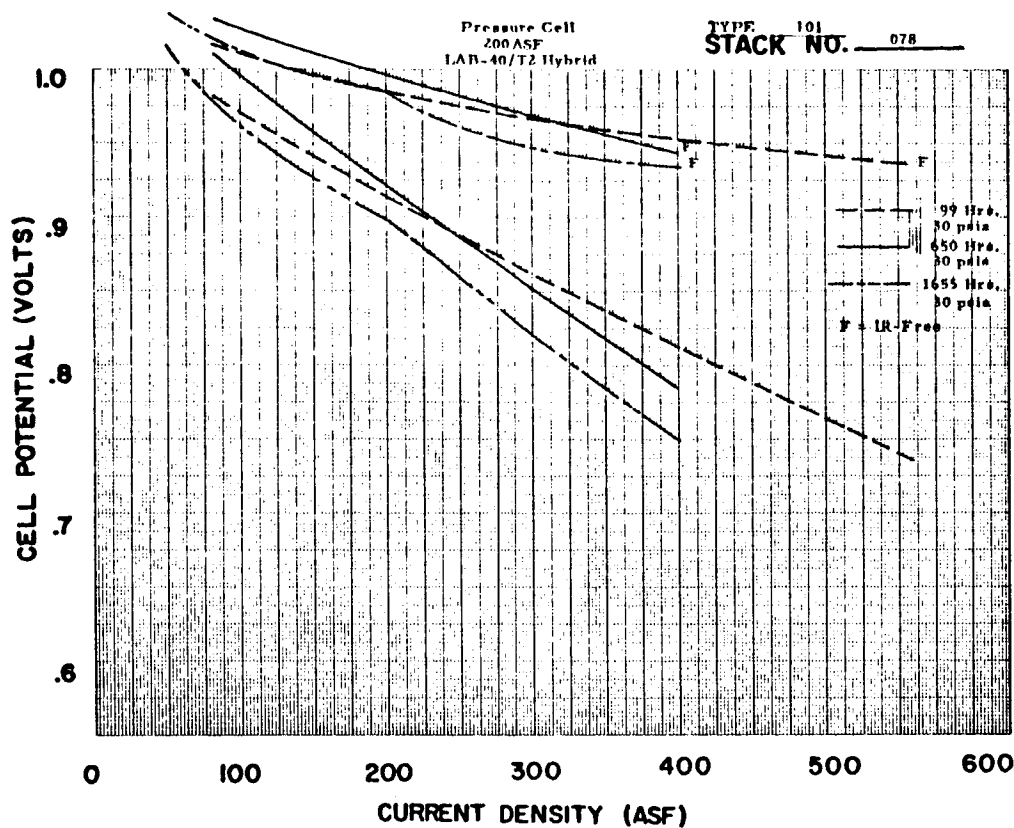
Fig. 109 - Polarization Curves - Stack A-078.

D-3562

200 ASF Test
LAB-40

TYPE 101
STACK NO. A079

22 Hrs.
536 Hrs.
1375 Hrs.

F = IR-Free

CURRENT DENSITY (ASF)
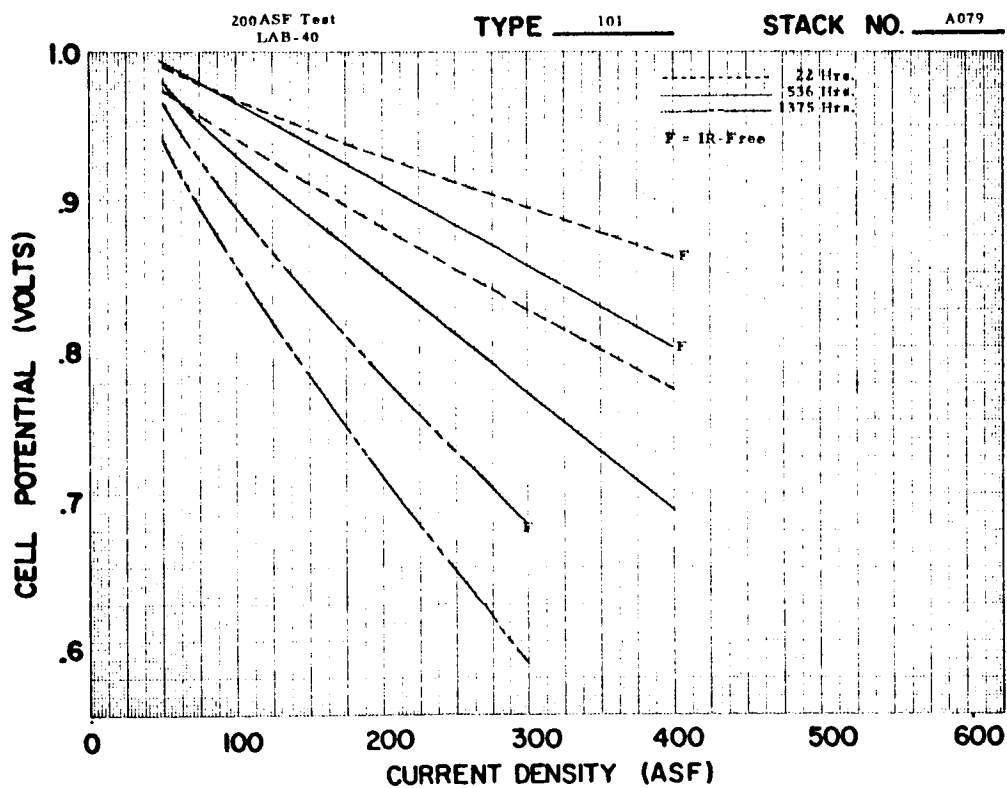
Fig. 110 - Polarization Curves - Stack A-079.

D-3563

-96-
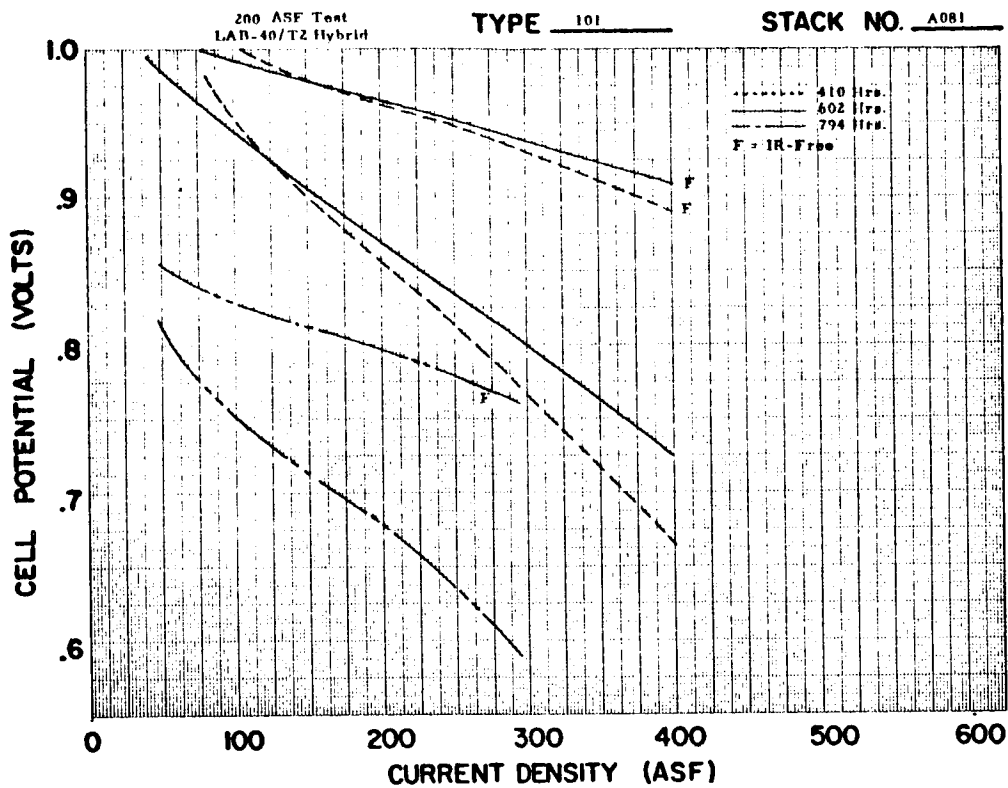
Fig. 111 - Polarization Curves - Stack A-081.
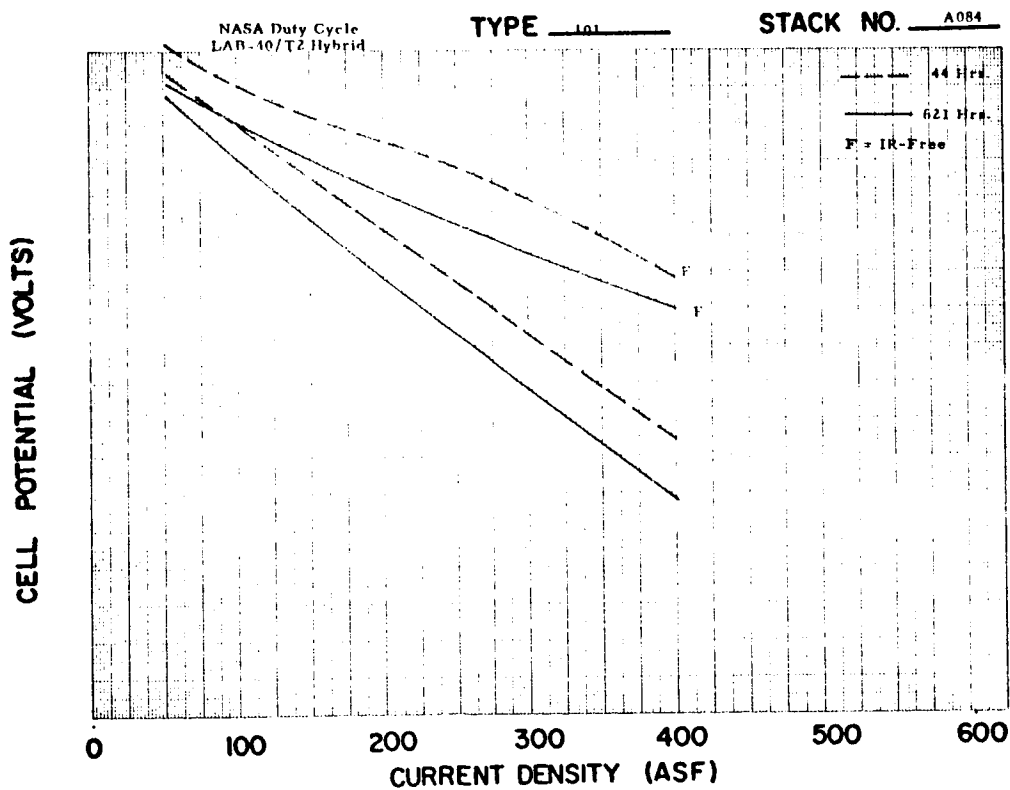
Fig. 112 - Polarization Curves - Stack A-084.
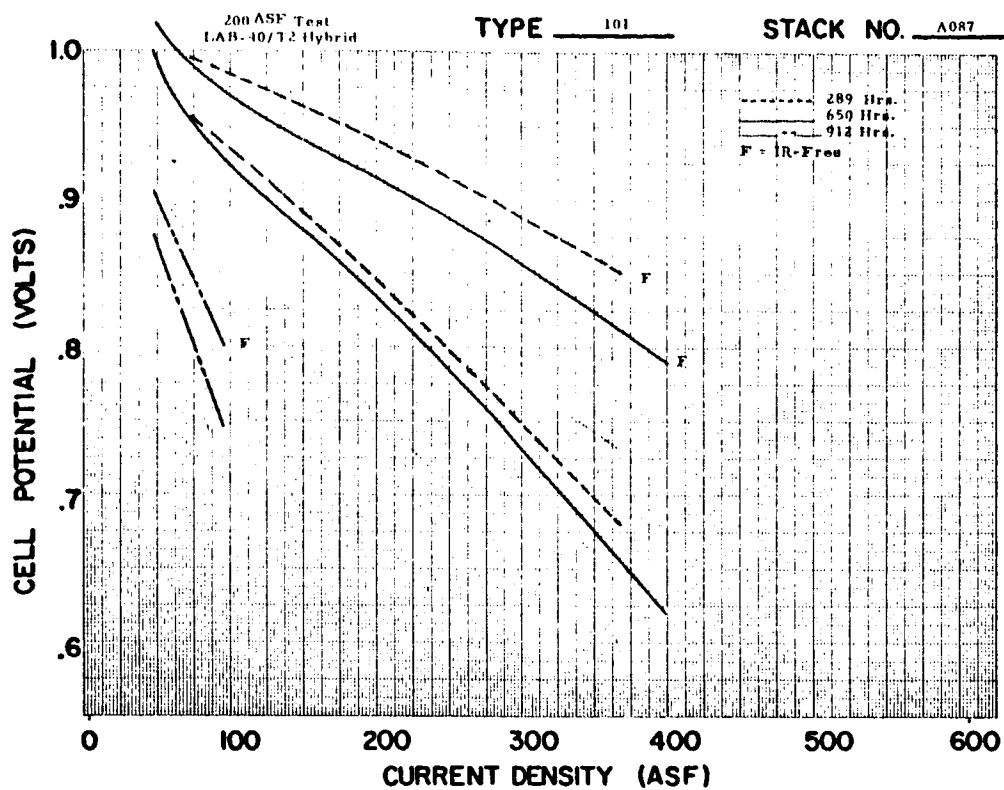
Fig. 113 - Polarization Curves - Stack A-087.

D-3566
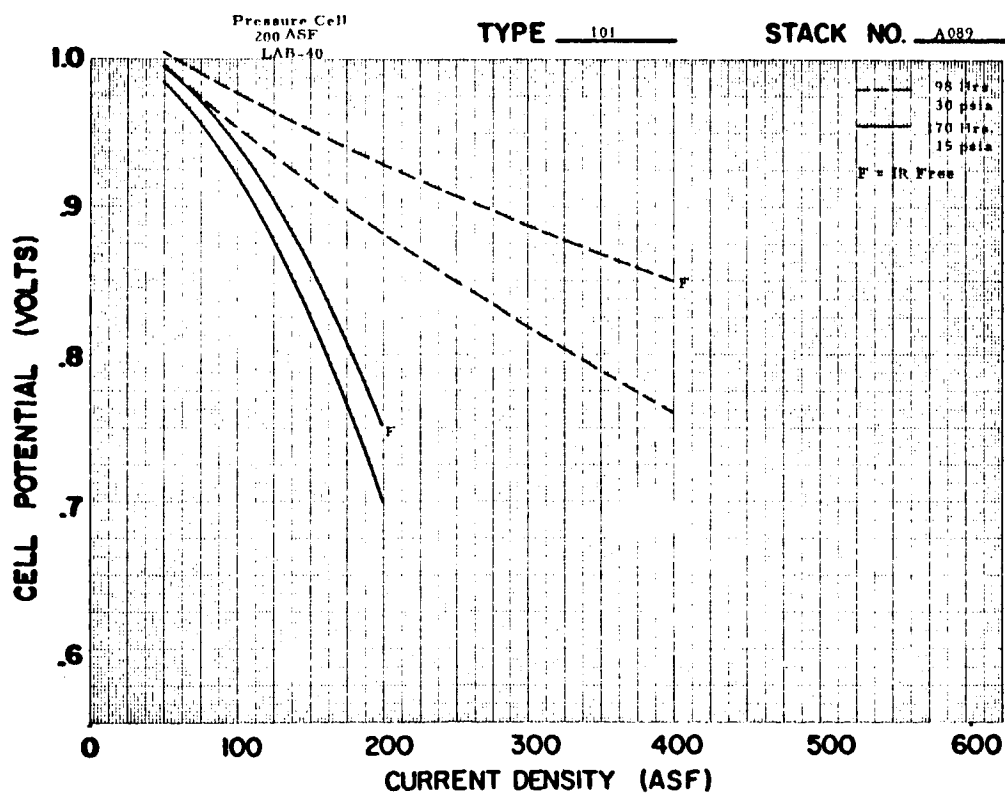
Fig. 114 - Polarization Curves - Stack A-089.

D-3567

-98-
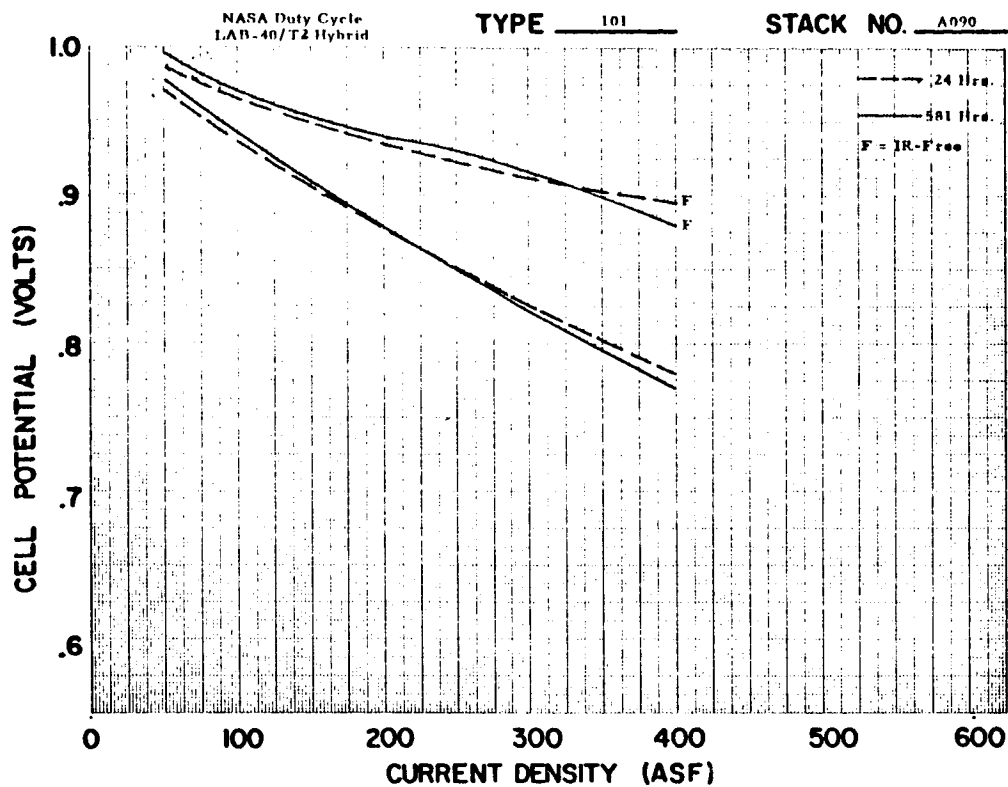
Fig. 115 - Polarization Curves - Stack A-090.
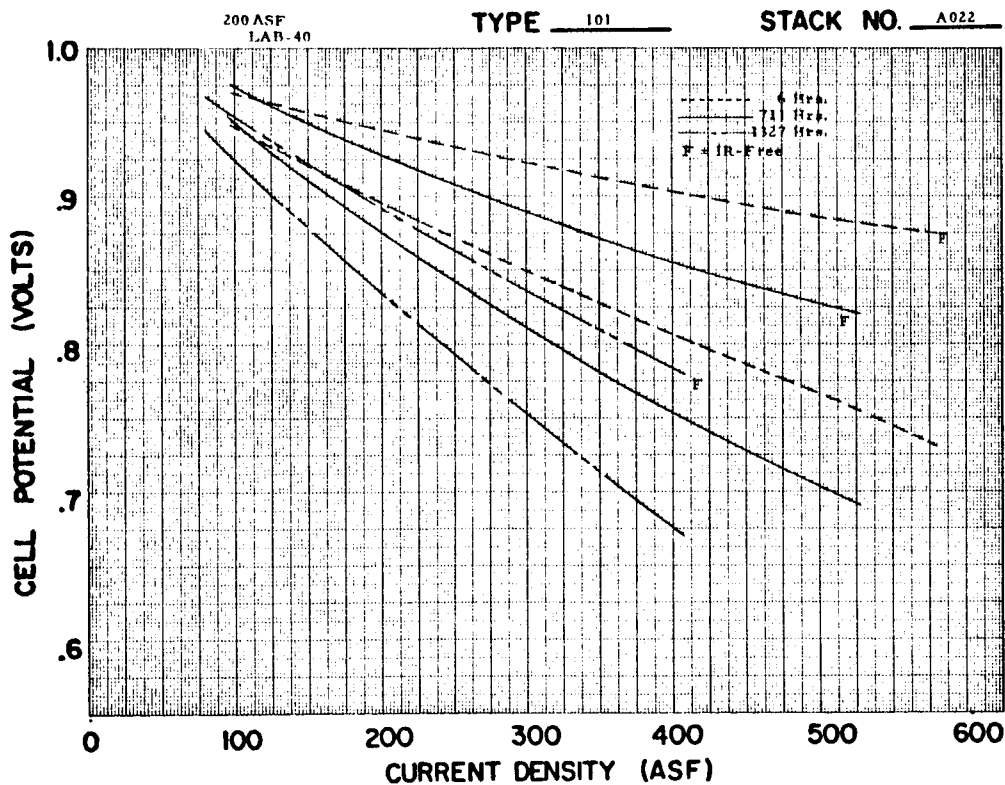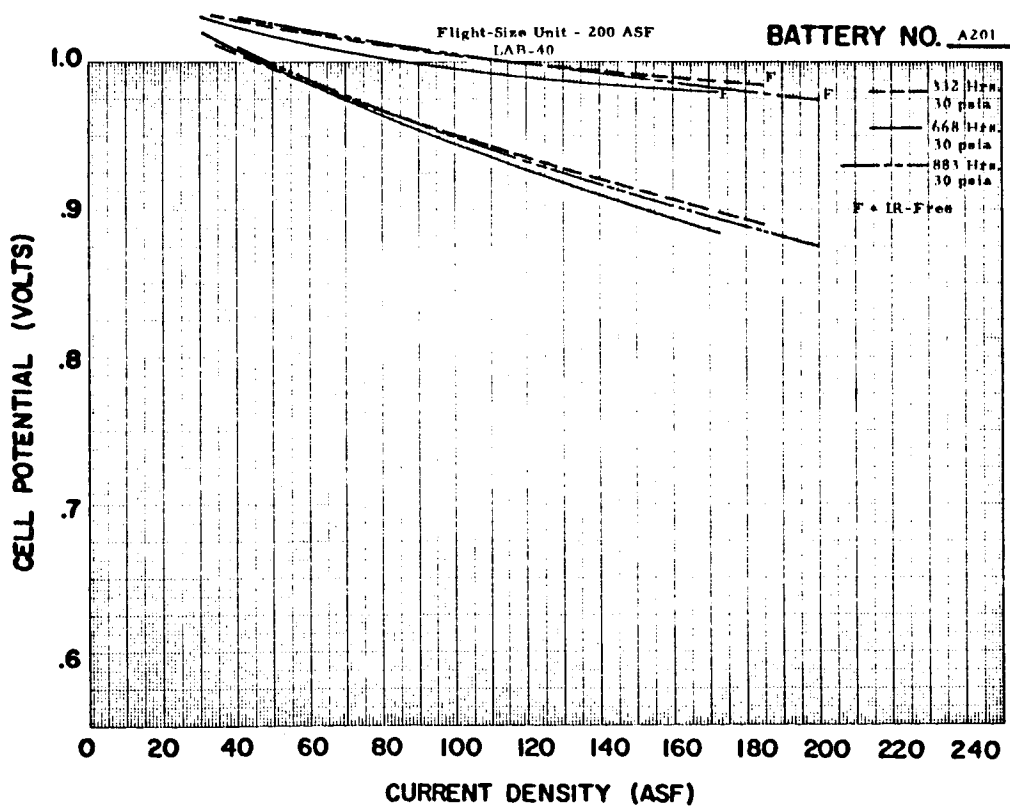

Fig. 116 - Polarization Curves - Stack A-201.

-99-
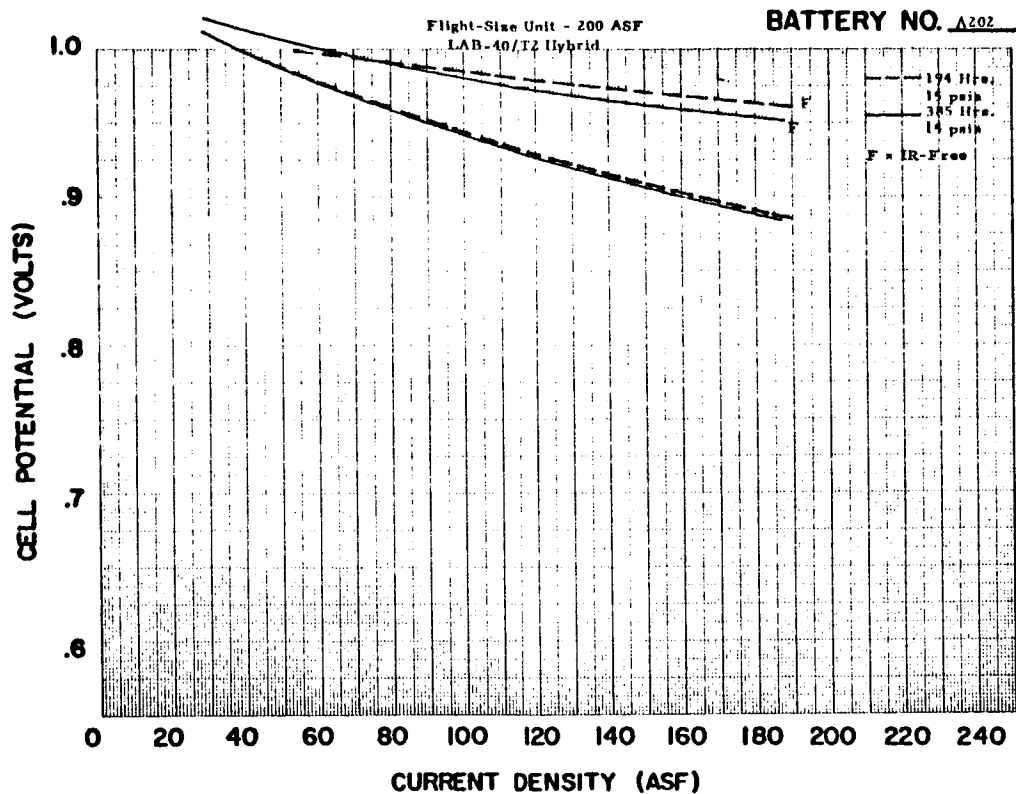
Fig. 117 - Polarization Curves - Stack A-202.
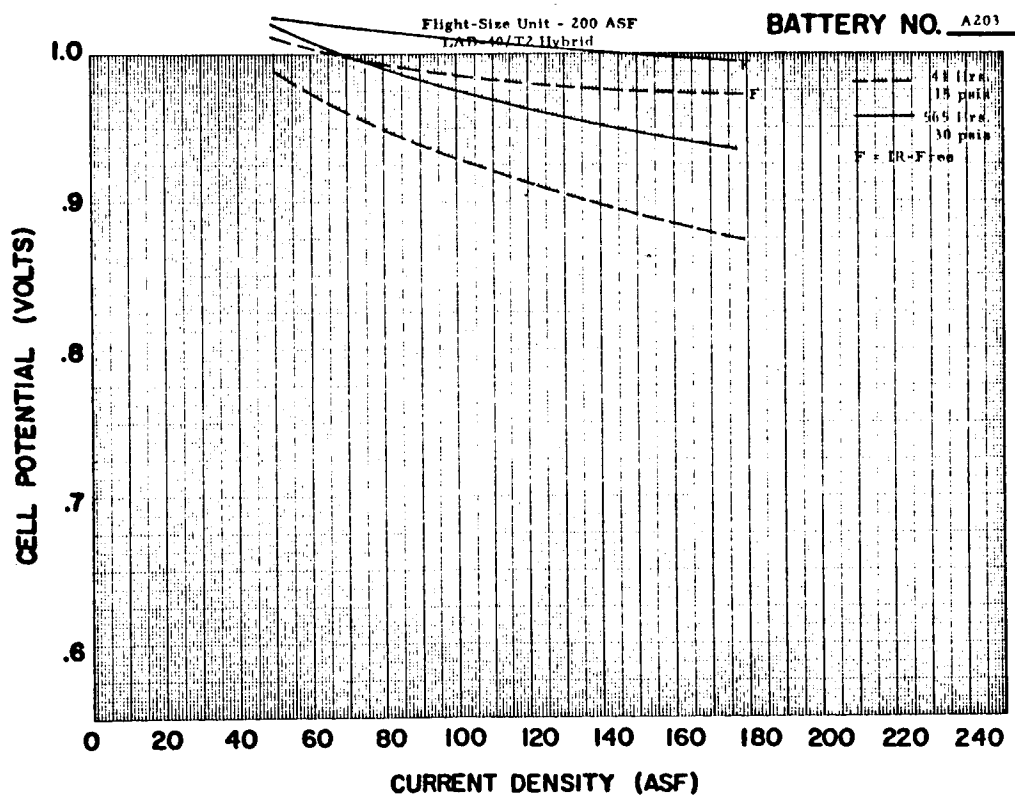
Fig. 118 - Polarization Curves - Stack A-203.

-100-
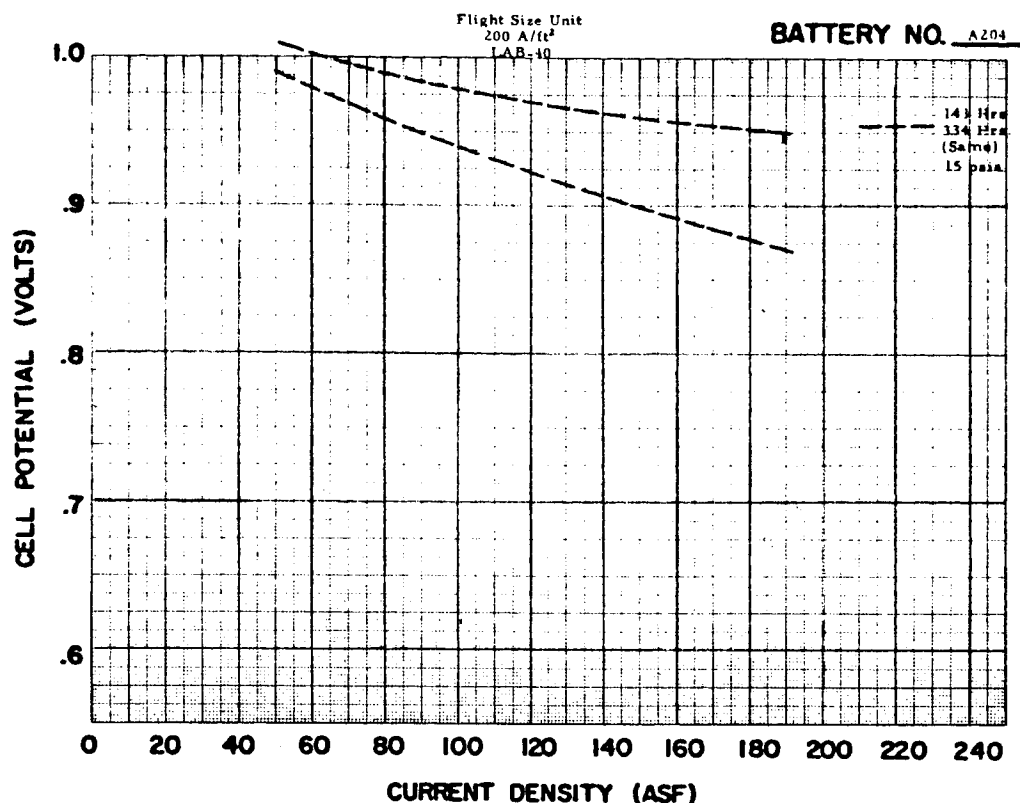
Fig. 119 - Polarization Curves - Stack A-204.

D-3572

3.3   Materials Compatibility.

Materials for use in cell fabrication are being tested in glass-covered Ni cylinders containing 200 ml of 12 N KOH. These cylinders are heated at 100°C for one week. Tested materials are then removed from the electrolyte, carefully washed and dried, and then investigated for any changes in physical properties. The electrolyte is examined for changes in surface tension, equilibrating molarity, and chemical contamination. From this entire group of samples tested, those which were clearly unacceptable because of physical deterioration were Epoxy No. 19 (Jones-Darney 5101, and Shell 871 with Z hardener); Epoxy Flex Bar X7050; EPT sheet compound; EPT "O" rings; expanded Teflon; polyurethane diaphragm; polyisoprene diaphragm, and Hypalon diaphragm.

Recent data, however, indicate that the low surface tension of the KOH has no deleterious effect upon cell life or performance.

Results on materials checked during this period are shown in Table IX.

TABLE IX
MATERIALS COMPATIBILITY TESTS

Material	KOH Surface Tension (dynes/cm)	KOH Normality	Weight Change (%)	Comments
Ni Crucible Bank	114.3	13.8	--	--
Air-Dried Neoprene Cement (B.F. Goodrich) on Polysulfone Sheet	114.6	12.9	-0.21	No obvious physical changes. KOH clear.
Epoxy No. 8-A: Maraset 124-C with No. 75 Hardener	115.5	14.2	-0.09	Ibid.
Ethylene Propylene Diaphragm AiResearch Division Garrett Corp.		13.1	-0.49	No obvious physical changes of material. KOH has tiny white particles suspended in it and had a rubberlike odor.
Ni Stranded Wire Welded to Ni Tab	114.0	13.9	0.00	Weld showed no sign of deterioration. Ni stranded wire corroded to a brownish color as of nickel oxide.
Styrene with 40% Fiber Glass Liquid Nitrogen Processing Corp., Malvern, Penna.	108.7	12.9	+4.41	Color changed from tan to white. Surface became rough. Swelling of ~7%. KOH clear.
Expanded Nylon, Code 10, 65-1 Stabilized Exmet Corp., Brdigeport, Conn.	105.6	12.8	-1.61	Postmortem of two NASA cells incorporating this material as KOH separator revealed its disintegration. These cells were on test ~1100 and 1800 hours. No indication of disintegration is evident from this one week test (168 hours). Material is stiff before test and no appreciable change in physical properties is detectable. Material did shrink ~12% across width and stretch ~4% along length, but it is possible to stretch material back to approximate original size. KOH clear. An extended time test is probably necessary to show effect of KOH upon this material.

TABLE IX (Cont'd)

MATERIALS COMPATIBILITY TESTS

Material	KOH Surface Tension (dynes/cm)	KOH Normality	Weight Change (%)	Comments
Expanded Teflon Exmet Corp. Bridgeport, Conn.	75.9	12.4	-0.28	Material more pliable than nylon. Material did shrink ~30% across width and stretch ~4% along length, but it is possible to stretch back to approximate original size. No obvious physical changes. KOH clear. Note low surface tension. No disintegration of this material has as yet been observed in any cell postmortems.
EPT Sheet Compound EP-47, Sulfur-free Philpott Rubber Co.	69.5	12.6	+0.55	Original whitish appearance Thyaran no longer evident after test. Sample became slightly sticky. No other obvious physical changes. Note low surface tension. KOH yellow.
Retested after washing, drying, and degreasing	109.8	14.0	-1.62	This sample of previously tested material was washed with Alconox, dried, washed with Freon, dried, and retested. Only notable change was that the surface tension of the KOH was not lowered as in the previous test. Physical properties of material remained unchanged. Slight stickiness still evident. KOH clear.
Sulfur-free EPT "O" Rings	60.9	13.6	-1.79	No obvious physical changes. KOH clear. Note low surface tension. Presoaking in KOH would probably eliminate this effect on surface tension.
Polyisoprene Diaphragm AiResearch Division, Garrett Corp.	71.0	12.9	-4.4	Material became soft and tacky. Lost physical strength Negligible thickness change. KOH clear. Unacceptable.

TABLE IX (Cont'd)

MATERIALS COMPATIBILITY TESTS

Material	KOH Surface Tension (dynes/cm)	KOH Normality	Weight Change (%)	Comments
Hypalon Diaphragm AiResearch Division, Garrett Corp.	83.0	12.7	-0.53	Material became stiff and lost much of its elasticity. Color changed from light blue to light green. No thickness change. KOH clear. Unacceptable.
Polyurethane Diaphragm AiResearch Division, Garrett Corp.	41.6	13.1	-40.4	Lost physical strength and could be easily pulled apart. Thickness decreased from 30 to 80%. Note low KOH surface tension. KOH slightly yellow. Unacceptable.
Premixed Epoxy (Short Glass Fill) Flex Bar X7050 U. S. Polymeric	55.3	13.5	+0.89	Physical appearance changed from a black to grey color. KOH adhered to surface and was not washed off during normal washing procedure. Material became soft and flakes off easily. KOH clear. Unacceptable.
Epoxy No. 19: Jones-Darney 5101 and Shell 871 with Z Hardener	99.7	14.0	+15.34	Material became very brittle and broke easily. Some internal cracking occurred during test. KOH clear. Unacceptable.
Silastic RTV 732 (Silicone Rubber) Dow Corning Corp.	104.0		-46.07	Tiny pinholes were formed in material and one edge became very thin. Very easily torn apart. KOH clear. Unacceptable.
Delrin (Acetal Resin Plastic) Mfgr: E. I. du Pont Five different types of material tested.				Material disintegrated. KOH yellow.

SECTION IV

4.0 FUTURE PLANS

4.1 Electrode Improvement.

Emphasis during the next period will be directed towards solving the major T-3 problems, and should include:

(a) Modifying the T-3 structure to permit better penetration of the active layer;

(b) Study the replacement of porous nickel by a more corrosion-resistant material (e.g., silver);

(c) Defining more exactly optimum active mix formulations with respect to type and amount of Teflon and extender, as well as other additives and mix consistency;

(d) Defining more exactly optimum fabrication parameters such as pressure and cure temperature and time.

4.2 Stack Testing.

Tests will be completed on those cells currently in progress. On the basis of these experimental results the best modification of the LAB-40 electrode will be selected for life testing at 200 ASF, both in hybrid cells with the T-2 anode and in cells using the LAB-40 electrode as both anode and cathode. Cells using T-3 cathodes and T-2 anodes will be factored into the life testing program as dictated by progress in those work areas outlined in Section 4.1 above. The testing capacity for pressure cells will be expanded.

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